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Testing Milk and milk products

FOR FAT AND TOTAL SOLIDS

Circular 630

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UNIVERSITY OF ILLINOIS • COLLEGE OF AGRICULTURE
Extension Service in Agriculture and Home Economics

COVER ILLUSTRATION

THE APPARATUS pictured on the cover is a mechanical caliper which was designed to standardize the reading of Babcock fat tests. It was invented by the late Julius Hortvet, chemist in the Dairy and Food Laboratory of the Department of Agriculture, Saint Paul, Minnesota.

The original apparatus was illuminated by an electric bulb thru an etched glass in the center with narrow mirrors, one on each side. The fat columns were read against the illuminated etched glass and the mirrors aided in leveling the line of vision with the calibration at the top and at the bottom of the fat column. The fat percentage was read by manipulating two blunt pointers, one being adjustable to the lower and the other to the upper extremity of the fat column by means of two knobbed screws.

This caliper was improved in the laboratories of the Vermont Agricultural Experiment Station (see reference 13, page 79). The combination etched glass and mirror was replaced with white opalescent glass so that the fat column could be read against a white background. The mirrors were discarded because the present bottles have marked lines three-fourths the circumference of the neck at each percent mark to aid in reading straight across the top and bottom of the fat columns. The blunt pointers on the original measuring device were replaced with adjustable needle points which add to the ease and accuracy of making fat readings. The small knobbed screws were replaced with larger ones to expedite raising and lowering the needle pointers. Finally a 5-inch reading glass was attached which magnifies the fat column about two and one-half times.

The time required to read tests is approximately the same as that with hand calipers, when one becomes accustomed to using the improved caliper. Fat readings for milk can be made with ease and precision to the accuracy of at least .05 percent. This apparatus is not available commercially at the present time but may be in the near future.

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TESTING MILK AND MILK PRODUCTS FOR FAT AND TOTAL SOLIDS

By E. O. HERREID, Professor of Dairy Technology

THE BABCOCK METHOD and its volumetric modifications for determining the fat content of milk and some milk products is one of the most important tests conducted in the dairy laboratory. Its simplicity has enabled persons with limited training to make fat determinations.

Unfortunately not all phases of the Babcock test or of the sampling procedures for the test have been standardized in the different sections of this country. Standardization is important for these reasons:

1. Dealers and handlers use the test as a basis for paying producers or marketing organizations for their milk.
2. Dealers and handlers also use the test as a basis for selling milk and cream and other dairy products, or for buying milk or cream or other dairy products from other dealers.
3. Knowledge is needed by plant operators of the fat content of their products in order to check the efficiency of their operations.
4. Variable methods lead to confusion and make it difficult, if not impossible, for any agency, public or private, to verify accurately either butterfat tests or the various other tests of milk and other dairy products.
5. Standardized methods are also important because milk and cream are shipped in interstate commerce.

The purpose of this publication is to describe testing procedures and technics which have given reliable results. In a few instances changes have been made in official procedures. These changes are justified because official methods^{1*} have lagged

* All superior figures with asterisks refer to literature citations on pages 79-80.

behind improvements in testing technics. This is not intended as a criticism of those responsible for the official methods — the fact is that the dairy industry in general has not demanded more refined technics for testing milk and milk products. It is hoped that this publication may help formulate regulations and standardize procedures for conducting the Babcock test and its modifications in Illinois and in other states as well.

To make this publication more useful to the dairy industry, directions for testing some of the common manufactured milk products are also included.

A discussion of the elementary physical and chemical properties of milk is given as a background for those who are engaged in testing work or supervising or who are training people for this work.

MILK CONSTITUENTS AND THEIR PROPERTIES

Milk fat. The fat in milk is present as small globules, which are suspended in the milk and form an emulsion. These globules are surrounded by phospholipids and proteins, which aid in stabilizing the fat emulsion.

Most milk globules are from 1 to 6 microns^a in diameter. One milliliter of milk may contain several billions of these globules, which vary in size according to the breed of the animal. The milk of the Jersey and Guernsey breeds has larger fat globules than that of the Ayrshire and Holstein breeds. In the milk of all breeds the fat globules gradually become smaller as the period of lactation progresses.

When milk is allowed to stand, the fat globules gather in clusters; these rise rapidly to form a layer of cream. At temperatures of 50°^b or lower, the fat in the clusters of fat globules is in a firm or solid state and will resist considerable agitation before there is much evidence of churning. If the milk is heated to 70°-80°, the fat is partially melted and is more sensitive to agitation. At these temperatures agitation will cause varying degrees of

^a It takes 25,400 microns to make an inch.

^b All temperatures are in degrees Fahrenheit, unless stated otherwise.

churning. Heating milk to 95° - 100° melts the fat and makes the emulsion more resistant to churning because the fat globules are less adhesive than at 70° - 80° . If the emulsion has, however, shown evidence of churning, liquid fat is apt to separate at 95° - 100° and this may interfere with accurate sampling. The physical conditions and distribution of the fat emulsion are important in obtaining representative samples for determining the fat content of milk.

Chemically, milk fat is known as a mixed triglyceride. It consists of a mixture of 18 fatty acids combined with glycerol. Some of these fatty acids are liquid; others are solid at room temperatures. It is this difference that accounts for some of the different physical characteristics of the fat emulsion during different seasons. Milk fat is relatively resistant to the action of strong acids, such as sulfuric. This property of milk is utilized in the Babcock test.

Milk fat contains yellow material known as carotene, which cows obtain from green feeds. Carotene is dissolved by the milk fat during digestion and assimilation. During the winter, milk fat gradually loses its yellow color because feed does not contain as much carotene when dry as it does when fed green in the summer.

Phospholipids. These substances are closely related to milk fat in their chemical composition. They include several compounds, such as lecithin, cephalin, and sphingomyelin. The phospholipids are attached chiefly to the surface of the fat globules, forming a layer around them. A part of this layer has a special attraction for the fat, while another part has an attraction for the watery portion of milk.

Phospholipids are the most important stabilizers of the fat emulsion in milk because they enable the globules, to a certain extent, to resist churning. Small amounts of the proteins in milk are also found with the phospholipids at the surface of the fat globules and have some stabilizing effect. When cream is churned, practically all the phospholipids are removed from the surfaces of the fat globules by the agitation and are found in the buttermilk.

Casein. This is the principal protein in cows' milk. It is present in milk as a suspension of very fine particles of calcium caseinate and calcium phosphate (a physical state known as a *colloidal dispersion*). Casein particles cannot be seen with the ordinary microscope. The shape of the particles cannot be seen even with the ultra-microscope. In milk that has been diluted with water, the particles can, with the ultra-microscope, be seen to reflect light. Because they reflect light, they contribute to the white appearance of milk. Casein can be coagulated by the natural development of lactic acid, by adding dilute mineral acids, alcohol, or rennet extract, which contains the enzyme rennin.

The average casein content of milk is about 3 percent but it varies with the fat content.

Lactalbumin. This protein makes up about .5 percent of milk. Its physical state in milk borders between true solution and colloidal dispersion. It is similar to albumin, the white gel-like substance in eggs. A small amount of lactalbumin is coagulated during pasteurization. Higher temperatures and longer exposures than are used for pasteurizing are necessary to coagulate all the lactalbumin. In the coagulated state, lactalbumin resembles the white of a boiled egg. In the manufacture of the common types of cheese, most of this protein is left in the whey.

Casein and lactalbumin are generally referred to as the proteins of milk. Lactoglobulin, the principal protein of colostrum milk, is present in normal milk but only in very small amounts. It decreases rapidly as milk becomes normal at the beginning of the lactation period. Another protein, euglobulin, is present in milk in very small amounts.

Lactose. Lactose, commonly called milk sugar, makes up about 5 percent of the content of milk. In the dry state, lactose has the same physical appearance as cane or beet sugar but is only about one-sixth as sweet. A pound of lactose has the same food value as a pound of cane or beet sugar. As this sugar is present in true solution (that is, it dissolves in the water of milk), it does not contribute to the color of milk.

Lactose is acted upon by certain bacteria and their enzymes

and is broken down to lactic acid. This fermentation is commonly called the souring of milk.

Milk salts. The mineral elements of milk are in the form of milk salts. These salts are, for the most part, in true solution. Altho they make up only a small part of the total content of milk (.9 percent), they are important in determining the reaction of milk to certain processing operations. They are also of great importance as human and animal food.

If a sample of milk is dried and heated to a high temperature in the presence of air, a white ash is obtained. This ash makes up about .65 percent of the milk. It contains calcium, magnesium, potassium phosphorus, sulfur, and small amounts of aluminum, copper, iron, and zinc. Because the milk is heated to high temperatures in the presence of air, the individual mineral elements are combined with oxygen in the ash and exist, for example, as calcium oxide, magnesium oxide, etc.

Milk also contains chlorides and iodides, but they are driven off at high temperatures and do not appear in the ash.

Enzymes. Normal milk contains several natural enzymes. So far as is known, lipase is the only one that may affect milk fat. Lipase is always present in cows' milk but is either more abundant or more active toward the end of the lactation period.

Lipase can break down milk fat. This is a slow process, but over a period of days enough fat may be broken down to lower the test of milk and cream. The activity of lipase produces an objectionable rancid flavor in milk.

Molds which sometimes are found growing on the surface of preserved composite milk samples also secrete an enzyme of the lipase type. This enzyme will lower the fat content of milk as much as .3 to .4 percent.^{11*}

Other components. Normal milk contains vitamins, leucocytes, other enzymes, and other substances in very small amounts, but these constituents are of no practical importance in determining the fat content of milk and milk products by the Babcock method and its modifications.

FACTORS THAT AFFECT COMPOSITION OF MILK

The average composition of milk and the range in its composition in this country is shown below. These figures are an average of the milk from cows of all breeds.^{9*}

<i>Constituent</i>	<i>Average percentage</i>	<i>Normal range in percentage</i>
Water.....	87.25	89.40 - 84.10
Fat.....	3.80	2.70 - 6.00
Protein.....	3.50	2.80 - 4.00
Lactose.....	4.80	4.50 - 5.20
Ash.....	.65	.60 - .70

The fat content of herd milk varies thruout the year, and tests should reflect that variation. If the test remains the same, the farmer has reason to suspect its accuracy. The fat content varies the most of all the solids, followed by proteins, lactose, and ash. This relationship holds true for individual cows as well as for breeds.

Breed. Milk varies more with breed than with any other factor. Breed is the main reason for variations in the composition of milk in different milk-producing sections of this country. In Oregon the average fat content is relatively high because the Jersey breed predominates; in Wisconsin it is relatively low because the Holstein breed predominates.

As the fat increases, the protein also increases but not so much. Milk from Jersey and Guernsey breeds therefore contains more fat and also more solids-not-fat than milk from the Ayrshire and Holstein breeds. The increase in solids-not-fat is due chiefly to the increase in proteins.

The amount of solids-not-fat in milk from the Ayrshire, Holstein, and shorthorn breeds varies from about 8.4 to 9 percent, and in milk from the Jersey and Guernsey breeds from about 8.8 to 9.5 percent.

Season. In May the fat content of milk goes down, staying at about the May level during June and July. About August it begins to increase, and it continues to increase during the cooler months. These seasonal changes come with changes in feed. A marked drop in solids-not-fat occurs with the high temperatures and dry weather of late summer, but these conditions do not seem to affect the fat, which decreases earlier in the season.

Interval between milkings. The longer the time between milking, the higher the milk yield and the lower the fat percentage. If the herd is milked at 5 o'clock in the evening and at 7 o'clock the next morning,

the time between the evening and morning milkings will be 14 hours and the fat content of the milk will usually be lower than in the next interval, which will be only 10 hours. The milking interval does not normally affect the average fat content of milk thruout the season.

Variations due to breeding. Sometimes the fat test and milk yield of a herd will decline over a period of years even tho feeding and management have been gradually improved. The cause usually lies with the transmitting ability of the herd sire. He probably has a number of low-testing daughters that have been kept in the herd. This may happen even when the herd sire has been carefully selected.

Breed population in the herd. In herds that consist of two or more breeds and cows of mixed breeding, the milk may have an average fat test of about 4 to 4.5 percent. The average fat test of the milk in such herds may fluctuate more than in herds that have only one breed because cows of different breeding may come into production at different times and thus lower or raise the average test of the herd.

Stage of lactation. This period, which lasts from calving until the final milking, is about 10 months long. The percentage of fat usually declines during the first or second month and then gradually increases until the end of the milking period. But under normal farm conditions the seasonal changes and changes in feed usually have more effect on the fat percentage than does the stage of lactation. After the third month of lactation, the solids-not-fat increase. This increase is due chiefly to an increase in the protein content.

Condition of cow at calving time.^{8*} A cow in good flesh at calving will give considerably more milk during her lactation period than a cow in poor condition, and the fat content will be higher.

Effect of gestation. Beginning about the fifth month of pregnancy there is a marked decline in milk yield until calving time. The fat percentage gradually increases during the period. Decline in production varies with different cows.

Effect of age. The composition of milk varies only slightly during the lifetime of a cow. Up to 12 years of age cows show a slight decline in the fat content of their milk. After about the eighth year the milk yield begins to decline.

Health of the cow. Mastitis causes radical changes in the composition of milk. Chlorides increase, as is indicated by a salty flavor and a lack of sweetness caused by a decrease in lactose. The fat and solids-not-fat decrease. The extent of the decrease depends on the severity of the infection. Other diseases cause production to decline.

The effect of health on the composition of the milk cannot be predicted. The fat content may increase or decrease or remain the same. Milk from infected quarters tends toward the neutral point and may

become alkaline, whereas milk from healthy cows is always acid in reaction. This change in reaction is the basis for the bromthymol-blue and other color tests used to diagnose udder infections.

Amount of milk and cream used at home. Milk to be used in the farmer's home may be taken from cans after it is cooled. As stirring devices are not usually available, milk at the top of the can, which is higher in fat content, is taken. This practice has the same effect as partial skimming and of course lowers the fat test.

FACTORS THAT AFFECT FAT CONTENT OF SEPARATED CREAM AND MARKET MILK

Farm-Separated Cream

The centrifugal separation of milk to obtain cream and skimmilk is a simple operation. The bowl in most separators turns 6,000 to 8,000 revolutions per minute. This creates a centrifugal force five thousand to six thousand times that of gravity. Skimmilk being heavier than cream (it has a specific gravity of 1.035 and cream, about 1), the centrifugal force is greatest on it. The skimmilk is therefore forced to the outside of the bowl and is discharged thru an opening above the dividing disk. The cream, being lighter, is forced toward the center, and as the bowl fills up, is discharged thru an opening in the dividing disk. The dividing disk prevents the separated cream and skimmilk from becoming mixed in the bowl.

Adjustment of cream or skimmilk screw. Every separator bowl has a screw which regulates the amount of cream or the amount of skimmilk which can be discharged from the bowl. The adjustment should be made to produce cream of 36 to 45 percent fat for these reasons: (1) higher percentages cause more fat to be lost in the skimmilk, and (2) the standard Babcock cream-test bottles (the only bottles approved in some states) will not estimate the fat in cream that tests more than 50 percent.

Speed of bowl. Increasing the speed of the bowl above normal increases the fat content of the cream. When the speed is below normal, the fat content is reduced and more fat is lost in the skimmilk. The speed of power-driven farm separators is usually constant except when power sources are overloaded. If the speed of the hand-driven machine is not uniform, the fat content of the cream can be expected to vary.

Fat content of milk. Milk from the higher-testing breeds will yield cream of higher fat content than that from the lower-testing breeds. This is true because the volume of cream obtained from milk containing 5 percent fat is only slightly greater than that obtained from 3.5 percent milk.

Rate of inflow. It is important to operate the separator according to the manufacturer's directions. Any change in rate at which the milk flows into the separator bowl changes the fat content of the cream. If the rate is reduced, the fat content of the cream will be higher than with a normal flow. If it is increased, the fat content will be lower; but if the flow is too fast, more fat will be lost in the skimmilk.

Temperature of milk. Lower temperatures give higher-testing cream. For example, milk separated at 90° yields cream that contains more fat than the cream from milk separated at 120°. But milk should not be separated at lower than 80° because such low temperatures are likely to cause more loss of fat in the skimmilk.

Deposits in bowl. While milk is being separated, a layer of material from the milk is deposited on the inside of the bowl. This layer gets thicker as the machine continues to be operated. Because a heavy deposit reduces the capacity of the bowl, it will lower the fat percentage of the cream. The smaller capacity of the bowl means that the milk will flow faster thru it; hence the centrifugal force will be applied for a shorter time and the fat percentage in the cream will be reduced.

Bowl deposits do not affect the fat percentage of cream if the farm separator is properly cleaned each time after it is used.

Amount of flushing. Some cream and skimmilk are left in the bowl after the separation is finished and may be forced out with either skimmilk or water. This dilutes the cream. The extent of dilution depends on the amount of flushing materials used. The same amount of skimmilk or water should be used each time for flushing, in order to prevent extreme changes in fat content.

Mechanical condition of separator. Vibration of the bowl during separation causes fat to be left in the skimmilk. Vibration may be caused by damage to the bowl or by worn or damaged bearings and gears. Worn disks reduce the capacity of the bowl and cause excessive losses of fat in the skimmilk.

Factory-Separated Cream and Market Milk

One of the crucial steps in testing milk and cream in milk plants is the obtaining of truly representative samples. Only by starting with such samples is it possible to know whether the

product does or does not comply with the fat standards set by city, state, and federal authorities.

Efficiency of agitator. A representative sample of milk or cream is the basis for obtaining an accurate test for fat. If a vat or holding tank that contains several lots of milk or cream has not been properly agitated before the sample is taken, the results are apt to be misleading.

To determine whether the milk is properly agitated, it is necessary to take samples at specified intervals and test them for fat. When three consecutive samples agree, it is safe to assume that the milk has been agitated long enough. Once the mixing efficiency of the agitator on a holding tank has been carefully determined, the results can be used as a guide for daily operations. This principle applies also in determining the agitating efficiency of pasteurizing vats for milk and cream.

Temperature at time of sampling. The temperature at which milk and cream are sampled is important in determining the fat content before pasteurizing and in making the calculations for standardizing. This factor has caused much difficulty in accounting for differences in fat content of milk bought and milk sold.

Operators sometimes take milk at low temperatures directly from the vat with a Babcock pipet, place it immediately in the test bottle, and determine the fat content for standardization purposes. To check the standardization process, they sometimes sample milk for the Babcock test by the same method and at a temperature near that of pasteurization. The results under these conditions will not be accurate. The Babcock test pipet holds

Everyone having to do with market milk and cream from producer to consumer needs to know the conditions and situations that cause the composition of milk and cream to vary. Only in that way will misunderstandings be prevented.

less weight of milk at 140° than it does at 40°. Milk pipetted into the test bottle at 140° will show .04 percent less fat than that pipetted at 100°, while at 40° the results will be about .08 percent higher than at 140°.

Variable results can be expected in the fat content of bottled milk if the temperature of sampling is not controlled in the standardization process. Furthermore, variable results will be obtained in accounting for milk fat if the milk is sampled at a different temperature from that of the milk sold.

Variation in sampling technics. Various sampling technics are used in daily plant operations. Patrons' milk from the receiving room may flow into large tanks, where it is held until enough has accumulated to begin processing operations. The time required to obtain a uniform distribution of fat in a tank depends on the degree of creaming, variation in fat content of individual lots of milk, and the temperature of the milk when it is delivered to the plant. Procedure for determining the agitating efficiency of vats and holding tanks is given on page 14.

In the high-temperature short-time method of pasteurizing, it is important to distribute the milk fat uniformly in holding tanks and to agitate it continuously until the milk is pasteurized; otherwise the bottled product is likely to vary in fat content.

Accuracy of standardizing process. It is often desirable to standardize milk and always necessary to standardize cream in order to comply with regulations for the different grades. Unless this work is carefully done, the fat content of the product is bound to vary. To do an accurate job, the pounds of product in the vat must be known, the sample to be tested must be representative, and the testing and calculating must be correct. When standardization is complete, the product should again be sampled and tested so that the accuracy of the process may be verified.

No accurate method of sampling milk delivered in tank cars and trucks. Altho it is frequently desirable to know the fat content of the milk delivered in railroad tank cars and tank trucks, no data have been published on methods of sampling and minimum number of samples required. Such information is

badly needed. In the first place it is desirable to verify the average fat test of milk received from the country plants and receiving stations. Furthermore, deliveries are often made by the same tank truck to several dealers, and these dealers want to know whether the milk they are receiving has the fat content specified in the invoices.

STANDARDS, DEFINITIONS, AND REGULATIONS

Federal Standards for Milk and Milk Products Used for Human Food^a

When milk and milk products are shipped from one state to another, they must comply with federal standards and those of the state into which they are shipped. For the most part, federal standards do not differ greatly from those in effect in the states. In fact when new federal standards are created or existing ones revised, they are almost invariably adopted by the states.

The standards published in 1944 for fat in the various dairy products are based on the Roesse-Gottlieb method for regulatory enforcement, as specified in *Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists*.^{1*} For some milk products, the analysis for total solids is also specified in this official publication. The standards published in 1936 do not specify the method for determining the percentages of fat and total solids.

Milk. Federal standards specify that milk shall be "the whole, fresh lacteal secretion obtained by the complete milking of one or

^a The standards for *milk*, *malted milk*, and *butter* are quoted from U. S. Department of Agriculture, Food and Drug Administration, S.R.A. 2, Rev. 5, November, 1936.

The standard for *homogenized milk* is from Ordinance and Code recommended in U. S. Public Health Service Bulletin 220, 1939.

The standards for *cream*, *skimmilk*, *evaporated skimmed milk*, and *sweetened condensed skimmed milk*; for *buttermilk* and *cultured buttermilk*; for *evaporated milk* and *sweetened condensed milk*; for *cheddar cheese*, *washed curd cheese* (soaked curd cheese), *Colby cheese*, *cream cheese*, *Neufchatel cheese*, and *creamed cottage cheese* are quoted from Federal Security Agency, Food and Drug Administration, S.R.A., F.D.C., 2, July, 1944.

The definition for *nonfat dry milk solids* was approved by act of Congress on March 2, 1944, and supersedes that given for dried skimmilk in the Food, Drug, and Cosmetic Act of 1938.

more healthy cows, excluding that obtained within 15 days before and 5 days after calving, or such longer period as may be necessary to render the milk practically colostrum-free." It must contain a minimum of 3.25 percent of fat, 8.5 percent of solids-not-fat, and 11.75 percent of total solids.

Homogenized milk is "milk which has been treated in such a manner as to insure break-up of the fat globules to such an extent that after 48 hours storage, no visible cream separation occurs on the milk and the fat percentage of the top 100 milliliters of milk in a quart bottle, or of a proportionate volume in containers of other sizes, does not differ by more than 10 percent of itself from the fat percentage of the remaining milk as determined after thorough mixing." [For example, if the upper 100 milliliters of milk from a quart, or upper 50 milliliters from a pint or upper 25 milliliters from a half pint, contains 4 percent of fat after 48 hours, the balance of the milk should not test less than 3.6 percent. The difference in test in this case would be .4 percent ($4.0 - 3.6 = .4$). This amounts to 10 percent of the total fat ($\frac{.4}{4} \times 100 = 10$).

Cream is "a class of food which is the sweet, fatty liquid or semi-liquid separated from milk, with or without the addition thereto and intimate admixture therewith of sweet milk or sweet skim milk. It may be pasteurized and if it contains less than 30 percent of milk fat, it may be homogenized. It contains not less than 18 percent of milk fat.

"Light cream, coffee cream, table cream, conforms to the definition and standard of identity prescribed for the cream class of food, except that it contains less than 30 percent of milk fat. Whipping cream is the class of food which conforms to the definition and standard prescribed for the cream class of food except that it contains not less than 30 percent of milk fat.

"Light whipping cream conforms to the definition and standard of identity prescribed for the whipping cream class of food except that it contains less than 36 percent of milk fat.

"Heavy cream, heavy whipping cream, conforms to the definition and standard of identity prescribed for the whipping cream class of food except that it contains not less than 36 percent of milk fat."

Skim milk (skimmed milk) is "that portion of milk which remains after removal of the cream in whole or in part."

Evaporated skimmed milk is "the product resulting from the evaporation of a considerable portion of the water from skimmed milk. It contains not less than 20 percent of milk solids."

Sweetened condensed skimmed milk is "the product resulting from the evaporation of a considerable portion of the water from

skimmed milk to which sugar and/or dextrose has been added. It contains not less than 24 percent of milk solids."

Nonfat dry milk solids, or defatted milk solids, is "the product resulting from the removal of fat and water from milk, and contains lactose, milk proteins, and milk minerals in the same relative proportions as in the fresh milk from which made. It contains not over 5 percentum by weight of moisture. The fat content is not over 1½ percentum by weight unless otherwise specified. The term 'milk,' when used herein, means sweet milk of cows."

Buttermilk is "the product that remains when fat is removed from milk or cream, sweet or sour, in the process of churning. It contains not less than 8.5 percent of milk solids-not-fat."

Cultured buttermilk is "the product obtained by souring pasteurized skimmed or partially skimmed milk by means of a suitable culture of lactic bacteria. It contains not less than 8.5 percent of milk solids-not-fat."

Evaporated milk is "the liquid food made by evaporating sweet milk to such point that it contains not less than 7.9 percent of milk fat and not less than 25.9 percent of total milk solids. It may contain one or both of the following optional ingredients:

"(1) Disodium phosphate or sodium citrate or both, or calcium chloride, added in a total quantity of not more than 0.1 percent by weight of the finished evaporated milk.

"(2) Vitamin D in such quantity as increases the total vitamin D content to not less than 7.5 U.S.P. units per avoirdupois ounce of finished evaporated milk.

"It may be homogenized. It is sealed in a container and so processed by heat as to prevent spoilage."

Sweetened condensed milk is "the liquid or semi-liquid food made by evaporating a mixture of sweet milk and refined sugar (sucrose) or any combination of refined sugar (sucrose) and refined corn sugar (dextrose) to such point that the finished sweetened condensed milk contains not less than 28 percent of total solids and not less than 8.5 percent of milk fat. The quantity of refined sugar (sucrose) or combination of such sugar and refined corn sugar (dextrose) used is sufficient to prevent spoilage.

"Such milk may be adjusted, before or after evaporation, by the addition or abstraction of cream or sweet skim milk, or the addition of concentrated sweet skim milk."

Malted milk is "the product made by combining whole milk with the liquid separated from a mash of ground barley malt and wheat flour, with or without the addition of sodium chloride, sodium bicarbonate, and potassium bicarbonate, in such a manner as to secure the full enzymic action of the malt extract, and by removing water. The

resulting product contains not less than 7.5 percent of butterfat and not more than 3.5 percent of moisture."

Cheddar cheese "contains not more than 39 percent of moisture and its solids contain not less than 50 percent of milk fat."

Washed curd cheese (soaked curd cheese) "contains not more than 42 percent of moisture and its solids contain not less than 50 percent of milk fat."

Colby cheese "contains not more than 40 percent of moisture, and its solids contain not less than 50 percent of milk fat."

Cream cheese (finished) "contains not less than 33 percent of milk fat and not more than 55 percent of moisture."

"In the preparation of cream cheese, one or any mixture of two or more of the optional ingredients gum karaya, gum tragacanth, carob bean gum, gelatin, or algin may be used; but the quantity of any such ingredient or mixture is such that the total weight of the solids contained therein is not more than 0.5 percent of the weight of the finished cream cheese. The name of the optional ingredients shall be indicated conspicuously on the label and immediately after the name 'Cream Cheese.'"

Neufchatel cheese (finished) "contains not less than 20 percent but less than 33 percent of milk fat and not more than 65 percent of moisture."

Creamed cottage cheese is "the soft uncured cheese prepared by mixing cottage cheese with pasteurized cream or a pasteurized mixture of cream with milk or skim milk or both. Such cream or mixture is used in such quantity that the milk fat added thereby is not less than 4 percent by weight of the finished creamed cottage cheese. The finished cream cottage cheese contains not more than 80 percent of moisture."

Butter is "the food product usually known as butter, and which is made exclusively from milk and cream, or both, with or without common salt, and with or without additional coloring matter. It contains not less than 80 percent by weight of milk fat, all tolerances having been allowed for." (*This definition is in conformity with the act of Congress approved March 4, 1923.*)

Standards for Milk Products Used for Animal Feed^a

As certain milk products may be used for animal feeds, standards are quoted for the guidance of dairy plant operators who may be contemplating the manufacture of these products. In all

^a The standards quoted here are from the official publication of the Association of American Feed Control Officials Incorporated, 1947.

standards for milk products, the Association of American Feed Control Officials regard lactic acid as part of the total solids.

Dried buttermilk (feeding) is "the product resulting from the removal of water from clean, sound buttermilk derived from natural cream to which no foreign substances have been added, excepting such as are necessary and permitted in the manufacture of butter. It contains not more than 8 percent of moisture, not more than 13 percent of mineral matter (ash), and not less than 5 percent of butterfat as determined by the Roesse-Gottlieb method." (*Adopted 1932*)

Evaporated, concentrated, or condensed buttermilk is "the product resulting from the removal of a considerable portion of water from clean, sound buttermilk derived from natural cream to which no foreign substances have been added, excepting such as are permitted and necessary in the manufacture of butter. It contains not less than 27 percent of total solids, not less than .055 percent of butterfat for each percent of solids, and not more than .14 percent of ash for each percent of solids." (*Adopted prior to 1928, amended 1944*)

Dried skimmed milk (feeding) is "the product resulting from the removal of water from clean, sound skimmed milk. It contains not more than 8 percent of moisture." (*Adopted 1930*)

Condensed skimmed milk is "the product resulting from the removal of a considerable portion of water from clean, soured skimmed milk. It contains not less than 27 percent of total solids." (*Adopted 1930*)

Dried cultured skimmed milk is "the product resulting from the removal of water from clean, sound skimmed milk which has been cultured by a suitable culture of lactic bacteria. It contains not more than 8 percent of moisture." (*Adopted 1932*)

Evaporated, concentrated, or condensed cultured skimmilk is "the product resulting from the removal of a considerable portion of water from clean, sound skimmed milk which has been cultured by a suitable culture of lactic bacteria. It contains not less than 27 percent of total solids." (*Adopted 1932*)

Dried whey is "the by-product from the manufacture of cheese or casein, either or both. This product shall contain at least 65 percent of lactose (milk sugar)." (*Adopted 1934*)

Condensed whey is "the product resulting from the removal of a considerable portion of water from clean, sound cheese or casein whey, either or both. It must not contain less than 62 percent of total whey solids. When this product contains less than 62 percent of total whey solids, it shall be designated "condensed whey, ——— percent solids." (*Adopted 1944*)

Dried whey solubles is "the dried product resulting from the re-

removal of albumen and the partial removal of milk sugar from clean, sound whey to which no foreign substances have been added except such as are necessary in the manufacture of milk sugar." (*Adopted 1944*)

Condensed whey solubles is "the product resulting from the removal of albumen and the partial removal of milk sugar from clean, sound whey to which no foreign substances have been added except such as are necessary in the manufacture of milk sugar." (*Adopted 1944*)

Dried whey product is "any dried product, not otherwise defined, composed of whey constituents and containing less than 55 percent lactose (milk sugar)." (*Adopted 1946*)

Casein (feeding) is "the product resulting from acid or rennet precipitation of skimmed milk. It must contain at least 80 percent of crude protein." (*Adopted 1946*)

Cheese rind is "cooked, partially defatted cheese rind." (*Adopted 1935*)

Suggested Definitions

Dairy plant. A dairy plant is any establishment owned by an individual, group of individuals, or a corporation where milk, cream, and other dairy products are bought and sold on the basis of their fat content and where the establishment is operated and licensed as prescribed by the proper regulatory official.

Regulatory official. The regulatory official is the representative of the commissioner of agriculture or the representative of any other official state or federal agency charged with the responsibility of administering regulations for the testing of milk, cream, and other dairy products, as may be designated.

Dairy license. A dairy license is a permit to sample and test milk and cream in any plant where these products are bought or sold on the basis of their fat or total-solids content, or in any laboratory where testing is done and the results are used as a basis for payment for milk and cream. The license shall be posted conspicuously in the place where licensee is employed.

Some Desirable Regulations^a

Obtaining a license. Anyone applying for a license to test dairy products must execute special forms as prescribed by the

^a These are at present (September 1946) suggested regulations which it is hoped Illinois and other states will officially adopt.

regulatory official at a predetermined time before the examination is desired.

To obtain a license, the applicant must have satisfactory vision for testing work. If vision is impaired, the proper corrections shall be made. He shall pass a satisfactory oral and written examination on a selected list of questions that apply to the sampling and testing of milk and cream and such other dairy products as are designated by the regulatory official. The applicant shall demonstrate his competence to determine accurately the milk-fat content of fresh and of preserved milk and cream samples and such other products as may be designated. The degree of accuracy shall not exceed the smallest division on an accurate test bottle.

After qualifying for a license, the applicant shall pay the stipulated fee. The license shall be valid for a specified period. It may be renewed provided the licensee has done satisfactory work, has passed examinations as required by the regulatory official, and has paid the renewal fee.

The license shall be revoked if the person is dishonest, incompetent, inaccurate, or careless. The revoked license shall be surrendered to the proper regulatory official.

Making records and reports. *Permanently bound book must be used.* The original record of a patron's delivery of milk or cream and the fat test shall be kept in chronological order in a permanently bound book. The pages of this book must be numbered consecutively and the book kept in the laboratory where the tests are made.

In this original record write each patron's name and number. Opposite the distinctly written name and number record the percentage of milk fat found in the patron's sample and the weight of the milk or cream. Make these entries with an indelible pencil or with pen and ink. When necessary to correct errors, draw a line thru the incorrect figure and place the correct figure on the same line. Have each entry on the original record (whether it is for one day or more than one day) dated and signed by the licensed person who made the fat determination.

When two or more licensed persons test samples in the same laboratory each must sign the records of the tests he makes.

If tests are made in a commercial laboratory that is not a part of the milk plant where the samples were taken, a carbon copy of the original laboratory records of the fat tests must be obtained and filed in the plant from which the samples came. All records of fat tests and the weights of patrons' milk to which the fat tests apply must be kept for at least one year or for such longer period as may be indicated by the regulatory official or his representative.

Records must be open to examination. All laboratory and plant records must be open to examination by the regulatory official or his representative. Any patron must be allowed to examine at any time the records of his fat tests and the weights of milk or cream he has delivered to the plant. On written request, a patron must be furnished a record of the fat percentages and weights of milk or cream which he delivered to the plant during a specified period.

Payment must be based on weight and fat content. Milk and cream delivered by patrons or by their representatives must be paid for on the basis of weight and fat content as determined by the Babcock method or by the Mojonnier ether-extraction method (a mechanized modification of the official Roesse-Gottlieb procedure).^{1*} Milk and cream sold by one dairy plant to any other plant for fluid, or for any manufacturing purpose whatsoever, must be paid for on the basis of weight and fat content. The invoice must indicate the weight of the milk, its fat percentage, and the total pounds of fat.

It is conceivable that, in the future, payment may also be made on the basis of the percentages of fat and of solids-not-fat, subject, of course, to the approval of the proper regulatory official.

Responsibility for observing regulations. The purchaser or receiver of milk or cream, or the licensed manager of any cream or milk-gathering station, manufacturing plant, or plant that receives or buys milk or cream from producers for sale or for resale or for manufacture, where the payment for milk or cream

is based in whole or in part on the milk-fat content, is responsible for any violation of regulations by any person who works under his direction or who is subject to his orders.

BABCOCK TEST FOR FAT IN MILK AND CREAM

Specifications for Equipment and Chemicals

Glassware for making test. Specifications for Babcock milk and cream test bottles and milk pipets have been adopted by the National Bureau of Standards, by the American Dairy Science Association, by the Association of Official Agricultural Chemists, and by a number of states. For a detailed description of Babcock glassware, refer to official procedures in the A. O. A. C.^{1*} The following bottles and pipet are specified for official use (see Fig. 1: A, B, C, D, F):

Milk test bottle

18-gram, 6-inch, graduated in .1% from 0 to 8%

Cream test bottles

9-gram, 6-inch, graduated in .5% from 0 to 50%

9-gram, 9-inch, graduated in .5% from 0 to 50%

18-gram, 9-inch, graduated in .5% from 0 to 50%

Milk pipet calibrated to contain 17.6 ml. at 68° F.

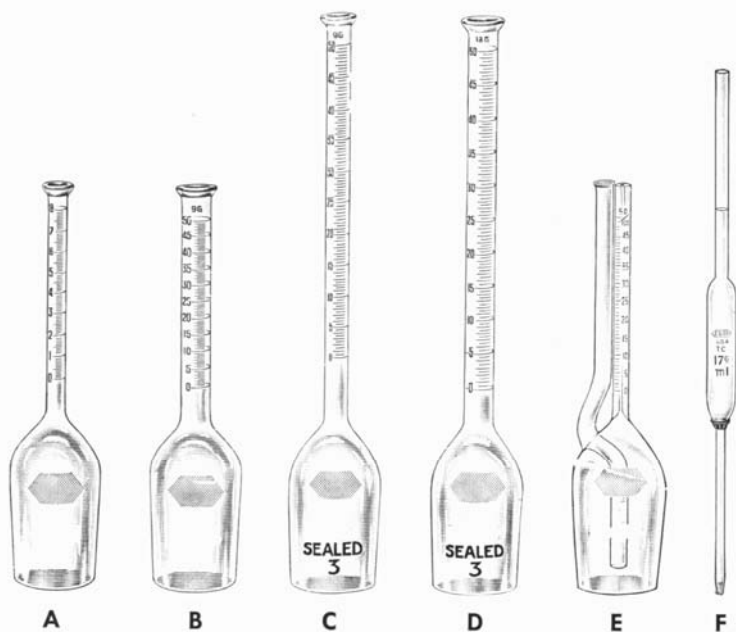
Specifications further provide that the maximum error of the total graduations in a bottle, or of any part of the graduations, shall not exceed the volume of the smallest unit of graduation. The pipet shall discharge its contents in 5 to 8 seconds. The maximum error of the pipet shall not exceed .05 ml.

Other types of cream test bottles are manufactured and used in some states. Individual bottles are calibrated from 0-30, 0-40, 0-50, 0-55, and 0-60 percent, with divisions varying from .2 percent to .5 percent. So far as the writer is aware, no comparison of the accuracy of these individual bottles for cream with the accuracy of the official ether-extraction method has been published. In design these bottles are like the official ones and are assumed to give as accurate results.

There are three types of cream test bottles that are not recommended because the graduations (1 percent) allow too much latitude in interpreting results; they cannot be read as accurately on individual samples as bottles that are calibrated in .5-percent

divisions. Two of these bottles are calibrated from 0 to 50 percent, and one from 0 to 55 percent.

Besides the 18-gram 8-percent bottle, two other milk test bottles are used in this country but neither is approved for milk by recognized official organizations. One bottle is calibrated from



Test bottles for milk and cream and a milk pipet. (A) Milk test bottle, 18-gram, 6-inch, graduated in .1% from 0 to 8%. (B, C, D) Cream test bottles graduated in .5% from 0 to 50%. B is a 9-gram, 6-inch bottle; C is a 9-gram, 9-inch bottle; and D is an 18-gram, 9-inch bottle. (E) Skimmilk test bottle, 18-gram, 6-inch, graduated in .01% from 0 to 50%. (F) Milk pipet calibrated to contain 17.6 ml. at 68° F.

(Fig. 1)

0 to 10 in .2-percent divisions and should not be used because it allows too much latitude in reading the test. The other bottle is calibrated from 0 to 10 in .1-percent divisions.

There are two bottles that are used for testing skimmilk, buttermilk, and whey, but neither is officially approved. Both are of 18-gram capacity. One is calibrated from 0 to .25. The other, which is preferable, is calibrated from 0 to .50 in .01-percent divisions (Fig. 1, E).

Manufacturers should be held responsible for the accuracy of the glassware used in testing. Each should be under bond to the state to calibrate and mark permanently each milk and cream test bottle and pipet with his name or trademark and the word *Sealed*.

Centrifuge. The centrifuge must be operated by mechanical power, preferably by electricity. The whirling chamber must be heated to an average temperature of 130° to 135° whether it has a full load of test bottles or less than a full load.

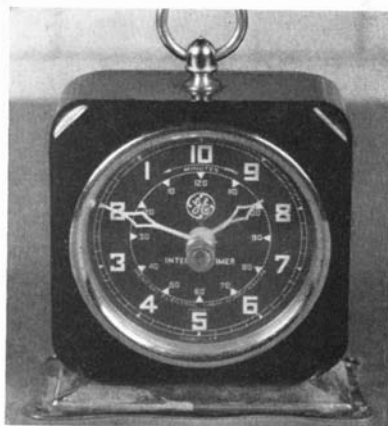
Experiments have shown^{19*} that the temperature in the whirling chamber of one electrically heated machine averaged at the outer circumference 136.5° and at the center, 131° . The temperature in the centrifuge must be carefully regulated because overheating will cause the contents of the bottles to contract when placed in the water bath. This may put the lower portion of the fat columns of the higher-testing milks below the graduated neck. Accurate readings cannot be made when the fat column has receded, because the meniscus (the curve at the top of the column) may become abnormally elongated, fat may stick to the glass, and the lower end of the fat column may become more rounded. Furthermore, excessive heating in the centrifuge will ruin the tests by forcing some fat out of the neck of the bottle. These conditions may be made worse by adding water to the test bottles at temperatures considerably above 140° between whirlings. Higher results are obtained from heated centrifuges than from unheated ones.^{10*}

The centrifuge (Fig. 2) must be equipped with an attached speed indicator (tachometer) and a thermometer. A thermostat is recommended. The machine must be set level, bolted securely to a firm table, and kept in the best mechanical condition.

The proper speed of the fully loaded centrifuge depends on the diameter of the wheel, as indicated in the following table:

<i>Diameter of wheel</i>	<i>Speed, r.p.m.</i>	<i>Diameter of wheel</i>	<i>Speed, r.p.m.</i>
12 inches.....	980	20 inches.....	759
14 inches.....	909	22 inches.....	724
16 inches.....	848	24 inches.....	693
18 inches.....	800		

The diameter of the wheel is the distance between the inside bottoms of opposite horizontal cups as measured thru the center of rotation.

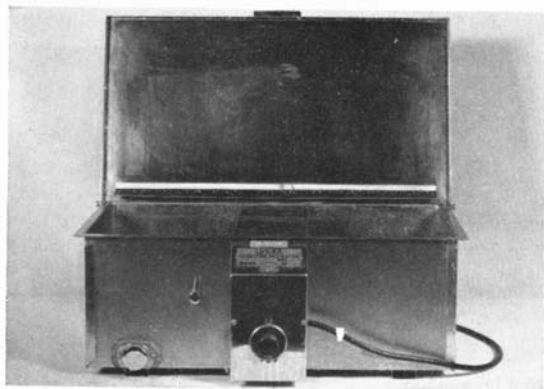
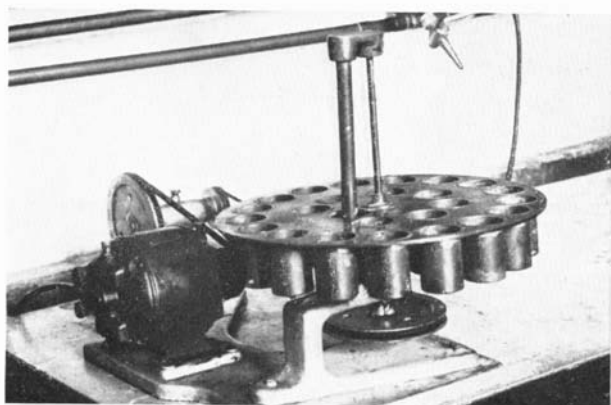


Alarm clock with automatic timing device. (Fig. 3)

Electrically operated centrifuge, with thermostat, speed indicator, and thermometer. (Fig. 2)

Mechanical test-bottle shaker.

(Fig. 4)



Water bath for tempering both samples and tests. (Fig. 5)

Time clock. To determine accurately the length of the whirling periods and the time the bottles should remain in the water bath, use an alarm clock with an automatic timing device (Fig. 3).

Babcock test-bottle shakers. In order to obtain the clearest fat columns, bottles must be shaken for 3 to 5 minutes as soon as acid is added. To standardize the shaking process, a mechanical shaking device (Fig. 4)^a that produces a rotary motion is necessary. Bottles can be placed on and taken off this machine while it is in motion.

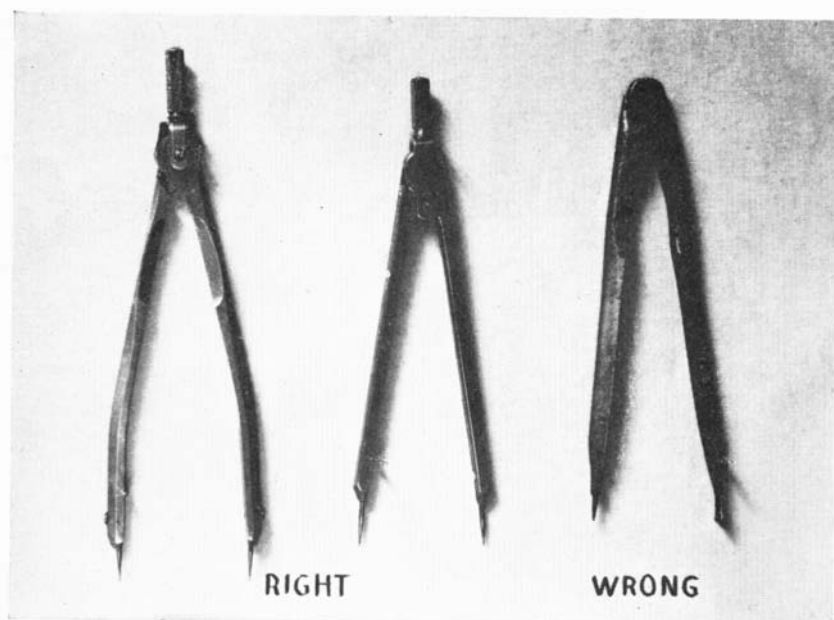
Water baths. *For adjusting temperature of samples.* Provide a water bath for adjusting and maintaining proper temperature of samples before they are pipetted. The bath should be equipped with separate compartments to keep sample bottles upright; it should have a thermometer that is accurate to 1° and an automatic heating element that will maintain proper temperature (Fig. 5).

For tempering finished tests.^b Have water bath equipped with a heater that will maintain a temperature of 135° to 140°, a thermometer that is accurate to 1°, and separate compartments to hold bottles upright. A thermostat is recommended. The purpose of the water bath is to standardize the temperature of the fat columns. Fat has a specific gravity of close to .9 at the time of reading. Necks of test bottles are calibrated to be read direct in percent when the fat has a specific gravity of .9 (Fig. 5).

Calipers (dividers). Various types of calipers are used for estimating the fat column. Some of these are superior and durable; some are inferior. A good caliper has a friction-locking device with an adjustment to compensate for wear, is resistant to corrosion, and has two sharp points. Three types of calipers are shown in Fig. 6.

^a This apparatus is called the "Burrell-Facile" test-bottle shaker and is made for either 24 or 36 bottles. It may be obtained from several well-known manufacturers of testing equipment.

^b Accurate readings can be made direct from the centrifuge when it is equipped with a thermostat and an electric heater that will maintain a temperature of about 130° to 140° F. in the whirling chamber, provided the room temperature is not less than 68° to 70° F. Testers are cautioned, however, to read their tests from a water bath, if it is so specified by state law.

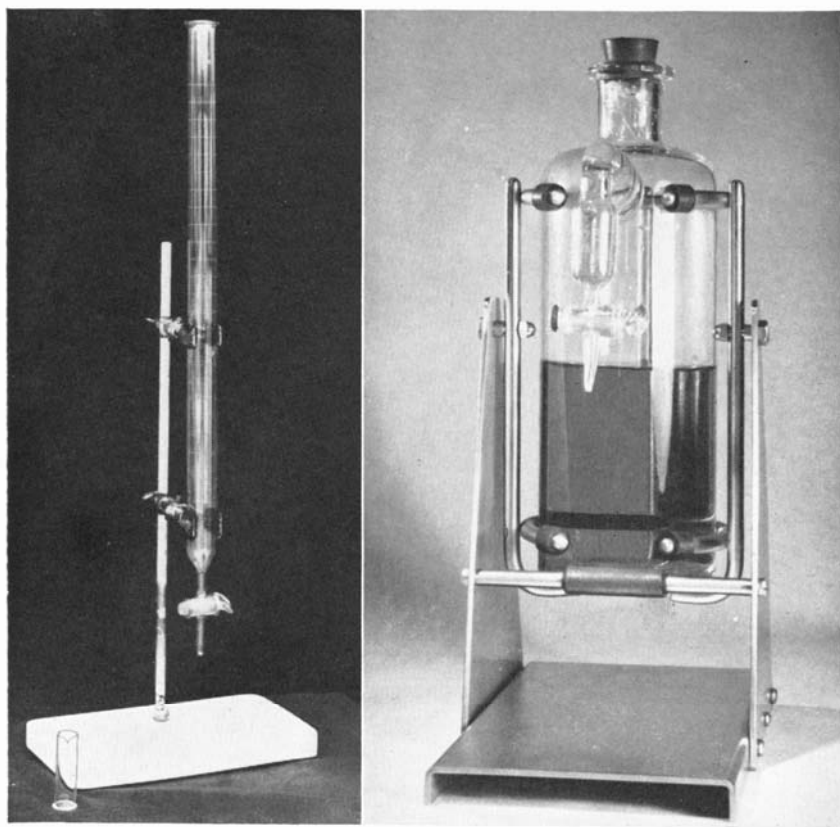


Calipers for measuring the fat column must be carefully chosen. The kind shown at the extreme left are best—note that the pointers are almost parallel when in position for reading the test. The center calipers have the same good features as those at the left except that the pointers are at a slightly greater angle when spread against the fat column. Calipers like those at the right should not be used—they do not lock, the pointers are bad, and the construction is not durable. Neither should fishtail calipers (*not shown*) be used. (Fig. 6)

Reading devices designed for more accurate measurements of the fat column are convenient and have been suggested by several investigators.^{13, 16, 32, 35*}

Hot-water facilities. Use distilled water or pure rain water to add to the test bottles. Keep this water in a noncorrosive container that is equipped with an indicating thermometer and with an automatic heating device that will hold the water at the correct temperature.

Acid dispenser. A graduated cylinder or dipper is a satisfactory dispenser when only a few tests are to be made. For a large number of tests a buret is more efficient. A buret attached to a glass bottle is also commonly used (Fig. 7).



Three common types of acid dispensers. (Fig. 7)

Sulfuric acid. Use acid that has a specific gravity of 1.82-1.83 at 68° F. This means that its purity is about 90 to 92 percent. Some lots of acid may be somewhat stronger. Babcock-test operators frequently report that a new shipment of acid is so strong that it burns the fat column. To correct this condition, use smaller amounts of acid to obtain clear and translucent fat columns, or correct the specific gravity of the acid by diluting it with water. (*As diluting acid takes time and is dangerous, it must be done only by a person who has some knowledge of chemistry. The more practical procedure is to use less acid.*)

Handle sulfuric acid with care. This acid will cause the skin to dry and crack and become sore. It will damage clothing, wooden table

tops, floors, and metal surfaces. If it is spilled on clothes, floors, or similar materials, neutralize it immediately with a mild alkali such as one of the common washing powders. *Do not use caustic soda or its commercial equivalent called lye.*

Store large carboys of acid in a room, preferably at 60°-70°, and keep them tightly stopped at all times. Acid takes water from air and loses strength. Sulfuric acid can be bought in glass-stoppered bottles with ten bottles to the case. This unit is more convenient to handle in the laboratory than the large carboys. The case and bottles can be returned for credit.

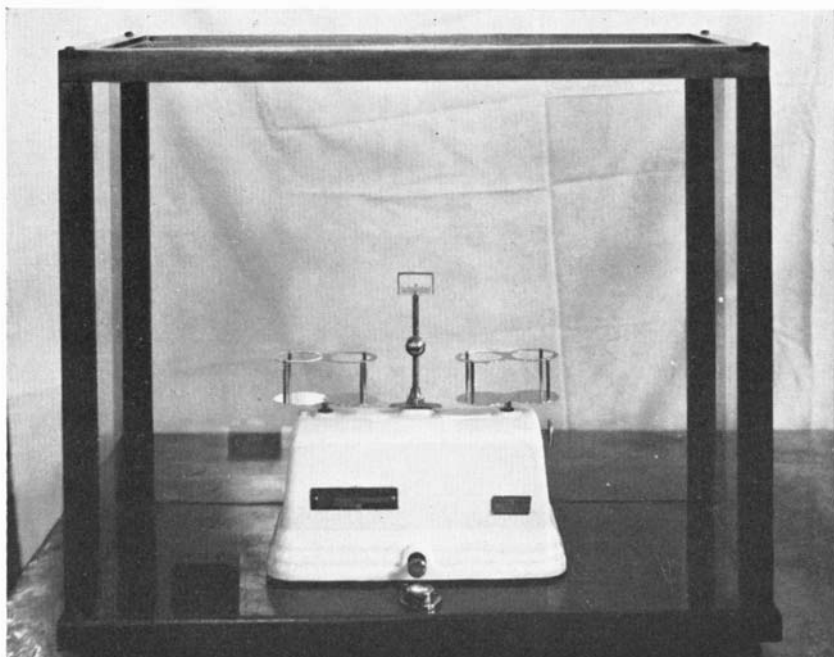
Meniscus remover (glymol). Just before reading the Babcock test for cream, add a few drops of glymol to the fat column to remove the meniscus (the curve at the top of the column). Glymol is a colorless mineral oil having a maximum specific gravity of .85 at 70°. To the oil has been added enough red dye to produce a sharp contrast with the yellow of the fat column.

Cream balances. Use a cream balance of single- or multiple-bottle capacity and a sensitivity of 30 milligrams. This sensitivity means that the pointer will move at least one division on the graduated scale when a 30-milligram weight is added to either pan while the scale has a full load and is balanced. Set balance level on a firm support that does not vibrate and see that it is protected from drafts (Fig. 8). Keep balance in good working condition, clean and polished, and away from excessive moisture and acid fumes.

Weights. Use weights made of durable material, such as gold- or chromium-plated brass, or stainless steel. See that the 9-gram weight does not have an error of more than 5 milligrams plus or minus, and that the 18-gram weight does not have an error of more than 9 milligrams plus or minus. After using the weights, take them off the scale and set them in their proper slot in a wooden block to protect them against mechanical injury. (*Do not set weights on a table where there is danger of contact with acids or other chemicals that will corrode them.*) Use weights that are plainly marked 9 or 18 grams.

Check weights at monthly intervals against weights known to be accurate. Handle them with forceps, not with fingers.

Sampling device. For sampling use a dipper, sampling tube,



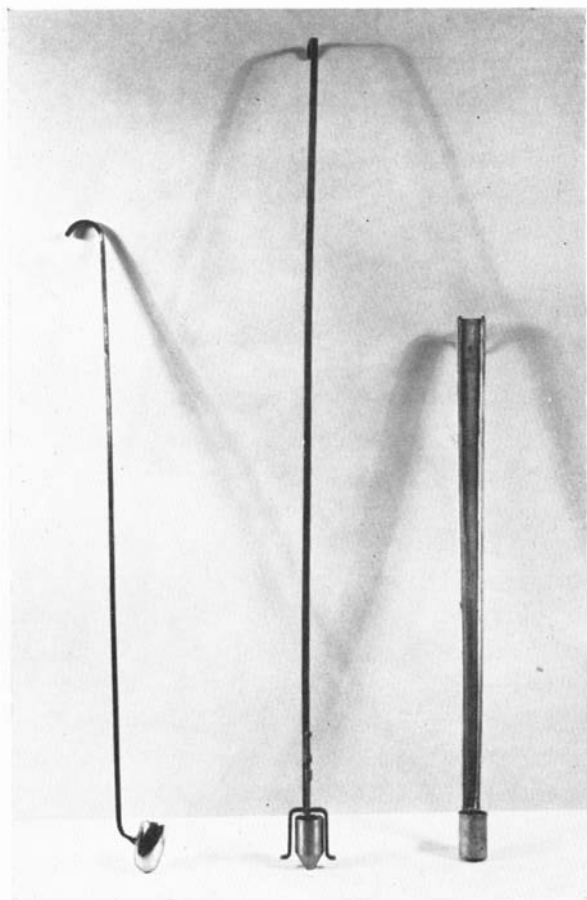
A cream balance is a very sensitive piece of equipment and should have especially good care. This balance is inclosed in a glass case 18 inches high, 24 inches long, and 10 inches wide. (Fig. 8)

or other approved equipment. Choose a device of the right size for the length of the sampling period. At the end of the composite period the sample must not be less than 140 ml. A fresh sample of milk or a sample of cream must not be less than 100 ml. Three samplers of the dipper type are shown in Fig. 9.

Stirring device. Use a stirrer that will produce uniform mixing. A good device is a rigid metal rod with a perforated metal disk 4 to 6 inches in diameter attached to the bottom. Whatever is used, it must be long enough to reach the extreme parts of the container.

Composite sample bottles. Bottles must be clean, dry, and round. They should be made of durable glass with shoulders sloped so that the fat which adheres to the glass may be dislodged easily. The bottle neck should have an opening $1\frac{1}{4}$ to $1\frac{1}{2}$ inches in diameter and a tight rubber stopper that is per-

manently attached to the bottle. A bottle with a metal screw cap or friction cap is not recommended unless it has a nonabsorbent gasket. The 8-ounce bottle is the smallest that can be used for composite samples.



Three sampling devices of the dipper type. (Fig. 9)

Each bottle must be numbered to identify one patron. This same number for this patron must be carried on the creamery records. The number should be engraved or countersunk on a brass plate. Each figure in the number should be at least $\frac{1}{4}$ inch high and at least $\frac{3}{16}$ of an inch wide. The indented numbers on the brass plate should be painted with durable black paint. The

number plate may be attached to the neck of the test bottle with copper wire or some other permanent attachment that is relatively noncorrosive (Fig. 10). Or it may be placed on top of the rubber stopper provided it is securely attached with two relatively noncorrosive cotter pins an inch long, one at each end of the plate. (*Do not paint numbers on rubber stoppers or on composite bottles, as they will gradually peel off.*)

Composite sample bottles in each plant must be numbered consecutively. Place date beside name and number of each patron when number is assigned and again when it is cancelled. Record in ink or in typewriting. File these records in the plant office and in the testing laboratory. Do not use two bottles with the same number unless they represent samples that are being taken in duplicate from the same patron. No bottle should carry two different number plates. When patrons leave a plant, do not reassign their numbers until the beginning of a composite period.

In order to destroy bacteria and molds that adhere to the glass and especially to the stoppers, wash, rinse, sterilize, and dry inner and outer surfaces of bottles and stoppers before using them again. To sterilize bottles and stoppers treat them in one of these three ways: (1) immerse them for at least 10 minutes in a chlorine solution containing 200 parts of chlorine per million; or (2) immerse them for at least 10 minutes in a diluted formalin solution that contains 4 parts of 40-percent formaldehyde solution and 96 parts of water; or (3) use some other disinfectant that is approved by regulatory officials. Do not use flowing steam or steam under pressure on bottles with rubber stoppers.

Have enough composite test bottles so that the samples, after they are tested, can be held in the plant for the length of time specified by the regulatory officials. Provide additional bottles of the same type for collecting and testing fresh samples as required by the regulatory officials.

Handling and Care of Composite Samples

Preservative. For each 8-ounce bottle, use one corrosive sublimate tablet (called mercuric chloride or bichloride of mercury) which contains .3 gram of mercuric chloride per tablet, or use any other equally effective preservative that is approved by the regu-



Approved type of composite bottle (*center*) and two kinds of cabinets. The bottle in the center is an approved type for either milk or cream. The stopper is permanently attached to the bottle and the customer's number (26) is countersunk on the brass plate immediately below the cap. On either side of the bottle are cabinets for storing composite samples. The cylindrical cabinet at the left is refrigerated with cold air. The box type of cabinet at the right is refrigerated with cold water. (Figs. 10, 11, 12)

latory officials. This amount of mercuric chloride will adequately preserve the milk sample during sampling periods of 7, 14, or 30 days and for the subsequent holding period as required by regulatory officials, provided the samples are properly refrigerated. If an equally effective liquid preservative is used, the amount added should not increase the final volume of the composite sample at the end of the period by more than .2 percent.

Put the preservative into a dry bottle. After each addition of milk to the composite sample bottle, rotate the bottle with one movement of the wrist in order to mix the layer of cream and the added milk. Do not shake the bottle as shaking tends to destabilize the emulsion of fat and interfere with accurate sampling.

Storing samples. Store preserved composite milk and cream samples during sampling period in a cabinet or box that is re-

refrigerated to maintain a temperature in the composite samples at 35° to 45°. The cabinet should be large enough to hold all sample bottles for the current sampling period. If composite samples are refrigerated with cold water or ice, the cabinet must be equipped with separate divisions which will allow the water to circulate, and also prevent bottles from tipping over. To keep unauthorized persons from tampering with samples, this cabinet must be locked with a durable lock. Not more than two persons in the employ of the plant or creamery should have keys to the composite sample cabinet. Each person having a key must be licensed to weigh, sample, and test milk and cream (Figs. 11 and 12).

Arrange sample bottles in a vertical position in cabinet or box in order according to the numbers that identify each patron, or on one or more trays to correspond to the loads from delivery trucks. Do not leave bottles outside refrigerated cabinet for more than one hour each day during sampling period. Keep stoppers tight in bottles at all times during storage.

Keep composite samples for 5 days after the day on which the tests were completed at the plant so that regulatory officials may, if they deem it necessary, determine accuracy of testing. Keep bottles tightly stoppered and refrigerated during this 5-day period.

Milk and cream samples must be tested for milk fat at the plant laboratory where the milk and cream are delivered by the patron. The proper regulatory official may, however, give written permission to remove fresh or preserved composite samples of milk and sweet cream to another laboratory to be tested for milk fat. If bottles are to be moved, pack them with their entire contents in a vertical position with cracked ice or other suitable refrigerant. The box or case must be sealed and locked by a person who is licensed to sample and test milk and cream.

Samples should be transported by a responsible carrier. Arrangement should be made to have samples promptly received and tested for milk fat by a licensed tester who has authority to break the seal. Seals on the boxes may be broken in transit by an authorized official to determine the fitness of the fresh and preserved milk and cream samples for testing. The official should

reseal the boxes and indicate date and time of inspection with his signature on a securely attached tag.

Preserved composite samples that show the growth of molds, evidence of churning, or other signs of improper care during the sampling period should never be used for testing. Instead, take approximately 150 ml. of a well-mixed sample of fresh milk from each patron whose composite sample is deemed unfit and use it as a basis for payment.

Composite samples to be tested for milk fat must be tested not later than the third day after the end of the sampling period.

Sampling Procedures^a

Composite milk samples. *When a patron delivers a large quantity of milk*, pour it directly into the weigh tank. If the pouring process does not mix the milk enough, an authorized regulatory official should indicate procedures or the installation of equipment, or both, that will insure uniform mixing of the fat. Remove from the weigh tank not less than 10 ml. of milk and deliver it directly into a composite bottle that contains the proper amount of preservative (*see page 34*).

If weigh tank is not large enough to hold entire delivery of a patron, weigh milk in about equal portions, mix well, and take one pint of milk from each portion. Mix these portions together and transfer not less than a 10-ml. sample to composite bottle as previously directed.

When a patron delivers one can or less of milk, sample the thoroly mixed milk directly by taking not less than 10 ml. for the composite sample. If milk is delivered in several small containers and there is not enough of it to be placed in weigh tank, pour contents of all containers into a can, stir thoroly, and sample as indicated above.

^a Some investigators^{22*} have shown that pouring milk into the weigh tank results in adequate mixing of the milk fat. Others^{3, 29*} have shown that variations occur at different points in the weigh tank, but that these variations can be prevented by stirring the milk before taking a sample.

Sampling from the weigh tank is sometimes complicated by the shape of the tank, the fat content of milk, the degree of creaming, the temperature of milk, and the season of the year. Manufacturers of dairy equipment could help solve this problem by constructing weigh tanks from which thoroly mixed samples of milk could be quickly obtained.

If milk and cream are received in same weigh tank, rinse cream out thoroly with water and drain tank well before milk is again weighed and sampled.

Rinse sampling dipper in thoroly mixed milk before taking out a portion for preserved composite sample. (The accuracy of the dipper method of sampling depends on two conditions: (1) a fair degree of uniformity in the amount of milk delivered daily by each patron; and (2) the fact that any change in the fat percentage of milk is gradual over any testing period except the decrease that occurs in early summer. If the amount of milk delivered by any patron varies greatly, the licensed tester or licensed manager should find the cause.)

Take not less than 140 ml. from each patron's deliveries during the sampling period, if the milk is normal. In taking composite samples use the same sampling device for all patrons of the plant.

Sampling abnormal milk. Provide a way to thaw frozen or partly frozen milk before sampling it. Do not sample milk that is curdled, churned, or partly churned. Give prompt written notice to any patron whose milk is delivered to the plant in condition unfit for sampling. In the notice state reasons why the milk is judged unfit and tell patron how to prevent the trouble. Record the dates of such deliveries.

Fresh samples. Fresh, unpreserved samples taken daily from a patron's deliveries can be used instead of preserved composite samples. A fresh individual sample must be representative of all the mixed milk taken from an individual animal or herd during a 24-hour period, and shall consist of not less than 150 ml. of milk. Hold sample in a properly numbered container, using the same kind of container as for preserved composite samples. Test fresh samples within 24 hours, and during this time hold them at 35° to 45°. The regulatory official shall have authority to order that the fresh samples be held for a designated period after they are tested.

Do not combine two or more fresh samples from a patron's deliveries, whether samples are preserved or unpreserved, when you are testing for fat.

When fat analyses of bottled milk from routes, stores, and other retail and wholesale outlets are desired, the untampered unit (half-pint, pint, quart, or two-quart) must be taken to the laboratory and mixed completely before sampling. If the unit is larger than two quarts, take only a pint sample from contents of the thoroly mixed unit and test it for fat.

Cream. Use same general procedures for sampling cream as for sampling milk. Because of the viscous nature of cream, more care is needed to get a representative sample. If testing daily, take not less than 100 ml. of cream from each delivery. Drain cream that sticks to sampling device from previous delivery as completely as possible and rinse the sampling device in the lot of cream which is to be sampled.

Preserved composite samples may be used for sweet cream. Such samples shall be handled in the manner prescribed for milk. Sour cream must be tested at time of delivery.

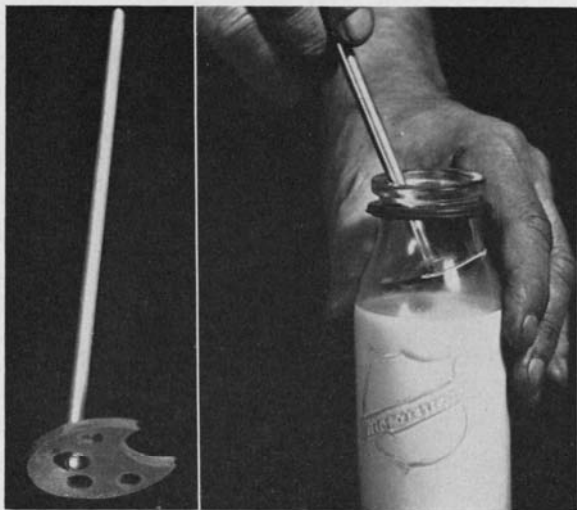
Abnormal cream. When a producer's cream is partly churned, soured, or lumpy, stir it thoroly before sampling. Heat frozen cream to 95°-100° and take sample immediately after melted fat has been evenly distributed. (Passing lumpy or sour cream thru a screen in an effort to get a more uniform sample will only make conditions worse.)

Procedure for Testing Milk

Preparing composite samples. Immerse composite samples in water bath to slightly above level of milk and heat to 95°-100°. Maintain water bath at 105° and control temperature automatically if possible. To prevent loss of contents and to keep water from getting in, keep bottles closed at all times except when they are being prepared for sampling.

If fat adheres to sides of bottle and to bottom of rubber stoppers, dislodge it with a wiping disk (Fig. 13) (a piece of flexible rubber about one inch wide attached to a rust resistant handle about 6 to 7 inches long). Scrape into warmed milk sample any fat that sticks to wiping disk. Do not use a brush because some of the fat may stick to the bristles.

Do not mix a sample by blowing air into it thru pipet. Distribute fat uniformly by pouring milk from original sample into mixing container four times. To keep from mixing air into the sample, pour milk against sides of container. This is especially important with homogenized milk, which foams more readily than does unhomogenized milk.



A rubber wiping disk is used to dislodge fat that has stuck to the sides of the bottle and the rubber stopper. This fat must be scraped into the warmed milk before the milk is drawn into the pipet. (Fig. 13)

Pipet sample immediately. Drain mixing container by placing it in an inverted position for at least 15 seconds before starting to prepare next sample.

Preparing fresh samples. Milk fat in fresh milk is not ordinarily hard to mix uniformly. Use same method and temperature for fresh samples as for composite samples. Pipet fresh samples at 95°-100° instead of at 60°-70° as specified by official method.^{1*} This higher sampling temperature is justified because Babcock method yields slightly higher results than Mojonnier method. The Babcock pipet will deliver slightly less milk at 95°-100° than at 60°-70° (see page 39).

Pipetting milk into test bottle. As soon as the sample is properly mixed, fill a 17.6-ml. pipet to above the mark on the draw tube. Do this slowly in order to prevent air bubbles. Place index finger on draw tube and adjust upper surface of milk to graduated mark by increasing pressure. Hold pipet clear of milk in container while making this adjustment.

To deliver charge, insert stem into neck of test bottle, as shown in Fig. 14. Note instructions given beside the picture.

Before removing a charge to be placed in a test bottle, always rinse the pipet in the sample by drawing it full of milk and then draining it. Also be sure that every test bottle is marked on the etched portion with the patron's number, preferably in white figures.



Correct way to drain milk from pipet into test bottle. Insert stem of pipet into neck of test bottle. Do not force milk out by blowing into pipet, but when active flow has ceased, allow milk to drain out as completely as it will for at least 10 seconds and then blow last drops from tip into test bottle. When testing a large number of samples, two pipets can be used. (Fig. 14)

Adding sulfuric acid. Have temperature of acid and milk at 68° - 70° . Vary amount of acid to whatever extent is necessary to give fat columns that are clear and entirely free from dark particles and undigested milk. This will require 15-17 ml. of acid. Add acid with small dipper or graduated cylinder, never with pipet. If a large number of samples are tested, a buret (Fig. 7) or a bottle that delivers multiple charges is most convenient. As you add the acid, hold test bottle at an angle and turn it to rinse down any milk that may cling in the neck.

As soon as acid is added, mix the contents. If only a few tests are to be made, gently rotate bottles for at least 3 minutes. If making a large number of tests, mix with a mechanical shaking

device. Set shaking device in motion before adding acid. With this device you need not shake bottles more than 3 to 5 minutes.

Do not shake bottles up and down. To do so may cause undigested particles of milk to obstruct neck of bottle; furthermore, the rapid chemical reaction produced may spray acid on workers and laboratory equipment. If any of the contents of any test bottle is lost in this manner, repeat the determination.

Centrifuging. Place bottles immediately in centrifuge. Balance them equally across the revolving axis. Use an even number of bottles.

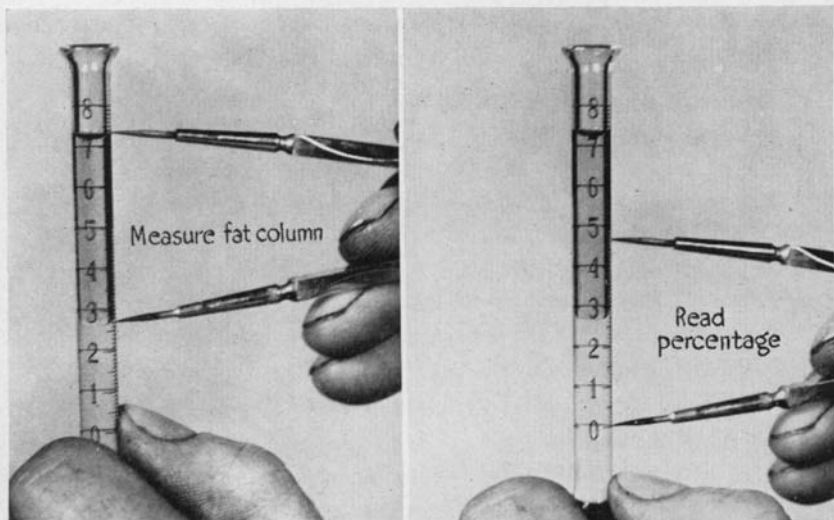
If making an odd number of tests, prepare another bottle, so that an even number can be whirled in the centrifuge. If only two bottles are whirled, and if trunnion cups are arranged in pairs, place each bottle in outer cup of opposite pairs to avoid a swinging motion that may break the bottle necks.

Whirl bottles for 5 minutes after machine has reached full speed. Stop machine and add water at 140° to about $\frac{1}{4}$ inch below base of neck.

Whirl bottles again for 2 minutes after machine has reached full speed; stop and add enough water at 140° to bring fat column within graduated neck. Do not allow tip of water dispenser to touch fat. Whirl again for 1 minute to collect the fat in graduated neck of bottle. Apply gradual pressure to brake to stop wheel, as sudden stoppage may splash some fat out of necks of test bottles.

Reading fat column. Immediately place bottles in a water bath at 135° - 140° for 5 minutes, with water level slightly above top part of fat column. Fat columns should be translucent, clear, and free of foreign materials.

Read tests in a well-lighted room. Before making readings, remove drops of water that cling to bottle neck. Hold bottles in a vertical position. Read the fat column from its lowest to its highest point—this includes meniscus (curved surface at top of fat column).^{1*} Measure fat column quickly with approved calipers (Fig. 15). After reading each bottle, place it again in bath to maintain proper water level.



Measuring the fat column. Use sharp-pointed calipers to read the fat column in the test bottle. Place lower pointer at extreme bottom of the fat column and upper pointer at upper edge of meniscus. Then using great care not to change the spread of the calipers, put lower pointer on zero mark. The upper pointer will then point to the fat percentage. Read to nearest tenth of 1 percent.

(Fig. 15)

Learn to read tests quickly and accurately. Slow reading may result in inaccurate tests because the fat column begins to move downward as soon as the bottle is taken out of the water bath.

For most purposes, one single test on each fresh or composite sample is enough. Duplicate tests may be made on samples that are being checked for compliance with standards and on others if there is some official question as to the fat content.

Record results. As each test is read, record fat percentage legibly after patron's number in a bound book. If test is of milk being processed, record percentage of fat as designated by the regulatory official.

Retests. If the fat reading of a composite sample of a patron's milk varies .3 percent or more from that of the preceding period, repeat the test immediately. Record results of final test to nearest tenth of a percent.

Make retests also if fat columns contain foreign materials, or if test has not been properly conducted.

Summary of procedure for testing milk

1. Heat samples to 95°-100°. Mix by pouring from one container to another.
2. Use an accurate 17.6-ml. pipet to measure charge of milk into an accurate test bottle. Cool to 68°-70°.
3. Add enough sulfuric acid at 68°-70° with specific gravity of 1.82-1.83 to give a clear fat column.
4. Mix acid and milk, preferably with a mechanical shaker.
5. Whirl 5 minutes.
6. Add water at 135°-140° to near base of neck.
7. Whirl 2 minutes.
8. Add water at 135°-140° to bring fat column within graduated neck of bottle.
9. Whirl 1 minute.
10. Place bottles in bath which is maintained at 135°-140° for 5 minutes with water level at 8-percent mark.
11. Remove from bath, read immediately, and record percent of fat.

Procedure for Testing Cream

Preparing sample. Place sample in water bath maintained at 105° and heat to 95°-100°. Keep water level in bath slightly above level of cream.

Balancing scales. Cream scales may be either torsion or beam type. Have bottles clean and dry on inside and outside and numbered the same as the samples that are weighed into them. Put bottles in bottle holders. Balance scale by adjusting a counterweight which usually is located on right-hand side or near top center. Lock scale when bottles are removed or added or when scale is moved from one location to another.

Mixing and weighing samples. As cream is more viscous than milk, it has to be mixed longer to distribute the fat uniformly. Pour samples from one container to another at least *six* times. Place a 9- or 18-gram weight on pan on *right* side. With a pipet add mixed cream to properly numbered dry bottle on *left* side to balance scales. Keep scales locked until most of sample

has been added. When it becomes necessary to add cream from pipet in drops, partly unlock scales to observe the balance pointer. Add cream until scales balance. (The reason for only partially unlocking the scale in first stages of weighing is to prevent either side of scale from dropping suddenly, as this might damage the weighing mechanism.)

In final stages of weighing each sample, unlock the scales completely to observe swinging position of pointer. If weighing is accurate, pointer, in one complete swing, should travel very nearly the same distance on each side of the zero point.

At this point in the weighing process, one bottle on the left side has a 9-gram charge of cream. Lock scales and remove weight. Mix another sample and weigh 9 grams of cream into properly numbered bottle on right side and in same way as before. Two samples have now been weighed. Repeat weighing procedure to full capacity of scales. Follow same procedure when using an 18-gram sample.

For transferring sample of cream to test bottle, use ordinarily a clean dry pipet. A single well-drained pipet is satisfactory if cream samples are in good condition and if their fat contents are within a range of 10 percent. If you use two different pipets, stand them in a vertical position to drain between successive weighings. If fat content of samples varies more than 10 percent, rinse pipet in water at 90° - 100° between weighings and allow to drain for at least 15 to 20 seconds. Rinse pipet in sample of well-mixed cream before removing a portion of the cream to be weighed.

Adding acid. Adjust cream in test bottle and the acid to about 68° - 72° . Acid may be added by either one of two approved methods:^{1*}

Method 1. Put 9 ml. of water at about 70° in each test bottle. Add 15.5 to 17 ml. of sulfuric acid, and shake contents promptly.

Method 2. Add 8-12 ml. of sulfuric acid for 9 grams of cream, and 14-17 ml. of sulfuric acid for 18 grams of cream. A good rule to follow with this method is to add enough acid to give the mixture a chocolate-brown color and then add water at 135° - 140° , using 4 to 5 ml. for the 9-gram sample and 8 to 10 ml. for the

18-gram sample. The water added slows the action of the acid. While adding acid and water, tilt and turn the bottles to release cream which clings to the neck. Promptly shake bottles, preferably with a mechanical shaker.

Centrifuging. Proceed as for milk (*see page 42*).

Reading fat column. Put test bottles in water bath at 135°-140° for 5 minutes with water level slightly above uppermost part of fat column. Before removing bottles from bath, add to each 3 to 4 drops of glymol or any other approved meniscus remover, and allow it to flow gently down inside wall of test bottle. (Do not add meniscus remover to more than two bottles in bath before reading them because it gradually mixes with fat and blurs separation line at top of fat column.)

Remove each test bottle from water bath, hold it in a vertical position, and measure fat column from its lowest point to the line which separates the fat from the meniscus remover. After reading each bottle, put it back in bath to maintain level of water.

Record results. As you read each test, record fat percentage after patron's number in a permanently bound book. Write clearly. If test is of cream that is being processed, record fat percentage in whatever way has been designated by the proper regulatory official.

Retests. Make retests if fat columns contain foreign materials, or if any part of test has not been carried out properly.

Summary of procedure for testing cream

1. Heat samples to 95°-100° in water bath maintained at 105°.
2. Place bottles, properly numbered, in holders and balance scale.
3. Mix samples and weigh them accurately into test bottles.
4. Add acid by one of two approved methods.
5. Promptly agitate bottles, preferably with a mechanical shaker.
6. Use centrifuge to complete steps in whirling, add water between whirlings, and place bottles in water bath in same way as for milk.
7. Before removing each bottle from bath, add meniscus remover and read fat direct in percent.

Appearance of Fat Column

The fat column of the Babcock test for milk and cream should be translucent (let some light thru), golden-yellow, and free from suspended particles.^{1*} When a column does not meet this standard, a tester should recognize the defect, know what caused it and how to correct it. These are the most common defects, and they are due to one or more of the causes listed:

Foam on top of column

Commonly due to carbonates in the water that was added when the sample was being centrifuged. Purer water is needed.

Discolored or dark particles in column

1. Too much acid.
2. Acid is too strong.
3. Milk or acid, or both, are too warm.
4. Sample was not mixed enough or mixing was delayed too long after acid was added.

Light-colored and white particles of fat in column

1. Acid is too weak.
2. Too little acid.
3. Milk or acid, or both, are too cold.
4. Acid and milk not thoroly enough mixed.

The fat columns of the modified Babcock tests should also be translucent and free from visible foreign materials.

Questions Concerning the Babcock Test

Why does milk pipet deliver 18 grams? The average specific gravity of fresh milk is about 1.032 when the temperature of the milk is 60° to 70° F. In this temperature range a 17.6-ml. pipet will deliver about 17.44 ml. of milk, as about .16 ml. clings to the inside of the pipet after the last drops are blown out. Therefore, the weight of milk delivered by the pipet is close to 18 grams ($1.032 \times 17.44 = 17.998$ grams).

In some states preserved composite milk is sampled at 95° to 100° F. At this temperature range the 17.6-ml. pipet will deliver an average of 17.91^{14*} grams of milk. This temperature of pipetting is preferable for both fresh and preserved composite milk samples because it results in closer agreement between the Babcock and Mojonnier methods.

Why is cream weighed into test bottle? There are three reasons why cream must be weighed and *not* measured into the test bottles: (1) Cream varies in weight with its fat content. As the fat content increases, a given volume weighs less. For example, a pipet full of 40-percent cream weighs less than the same volume of 20-percent cream. (2) The viscosity of cream increases with its fat content. This means that more 40-percent cream than 20-percent cream will stay in the pipet. (3) Because the viscosity of cream fluctuates, cream will contain varying amounts of air. The more air, the less a given volume of cream will weigh.

What other precautions are needed in testing cream? See that the sample is well mixed. Be sure the scales are accurate and protected from drafts. Have the test bottles clean and dry. Weigh the samples at room temperature, preferably about 70°-72°.

Why can fat in test bottle be read direct in percent by weight? Testers often ask why the Babcock test bottle is read by *volume* and the fat content is reported in *percent by weight*. To understand why this can be done, we need to consider the specific gravity of the fat and the volume capacity of the graduated portion of the bottle neck. We may assume that we are using a test bottle that is designed to take 18 grams of milk, and that this amount of milk has been placed in it. The neck of the bottle has been so marked off that each major division will hold .2 ml. of fat. The specific gravity of the fat when the test is being read at 135°-140° is approximately .9. Since the specific gravity of the fat is .9, each .2 ml. weighs .18 gram (.2 ml. \times .9 = .18). Since .18 gram is $\frac{1}{100}$ of 18 grams, each major division measures 1 percent of the weight of the milk in the test bottle. By the same reasoning, each .02-ml. division on the neck of the bottle measures $\frac{1}{10}$ of 1 percent of fat by weight.

Why is neck of bottle marked to take 8 percent of fat when milk does not often contain this amount? This is done so there will be no danger of the contents overflowing when the water is added. The full capacity of the neck should never be counted on, for it is difficult to standardize conditions so that all

the fat can be collected within the calibrated neck at the time of reading. The same is true of cream and skimmilk test bottles.

What is purpose of sulfuric acid? Sulfuric acid hydrolyzes the solids-not-fat and destroys the emulsion. The heat generated in this process liquefies the fat so that it can be easily separated from the acid solution. The sulfuric acid also increases the difference between the specific gravity of the fat and the rest of the solution, so that the fat will collect more quickly in the neck of the bottle when centrifugal force is applied.

Why add hot water between whirlings? The hot water keeps the fat in a liquid condition, washes it of impurities, and forces it into the graduated neck of the bottle. It also serves as a clear liquid onto which the fat can set apart from the dark dissolved materials in the bulb of the bottle.

Why is temperature of bath important? It is important because the bottles are calibrated direct in percent at 135°-140°. Extreme fluctuations in temperature of water bath will cause errors in the fat reading. The fat in the column has an expansion coefficient of .0007558.^{18*} A difference of 36° F. (20° C.) in the fat column would make a difference of .06 percent in the fat reading of milk that contains 4 percent of fat. This is illustrated in the following calculation:

.8 ml. = volume in bottle neck occupied by fat from 4-percent milk

.0007558 ml. = expansion coefficient per ml. of fat for each degree Centigrade

20° C. = assumed difference in temperature

1.6 ml. = capacity of neck of test bottle

8 percent = total graduation on neck of bottle

$.0007558 \times .8 \times 20 = .01209$ ml. fat

$\frac{.01209}{1.6} \times 8 = .06$ percent.

Why is meniscus included in reading of Babcock test for milk? The meniscus is included for the milk test because some fat remains in the bulb of the bottle and is not forced into the fat column.^{2, 10*} The Babcock method, however, gives a higher test for milk than does the Mojonnier method.

The meniscus was specified to be removed before reading the Babcock test for cream so that the results would agree more closely with those obtained by the official method. Nevertheless the Babcock method gives slightly higher average results on cream than does the Mojonnier method.^{15*}

Should tests made by milk plant and by dairy herd improvement association testers agree? These tests may not always agree for these reasons: (1) The number of tests over a given period are not always the same. The farmer's milk is usually tested weekly or semimonthly at the creamery; while in the dairy herd improvement association, samples of the composite milk from two consecutive milkings may be tested monthly or even every two months. (2) The dairy herd improvement association test is of only one day's production, and so would be subject to the fluctuations that occur from day to day in both fat percentage and yield of milk. The creamery test is made of a composite sample from the deliveries of one to two weeks, and so may be higher or lower than the test of any one day's delivery during this period. (3) Milk plants usually are required to have modern laboratories where it is possible to do precise work. Dairy herd improvement association testers do not always have the best equipment. For example they are likely not to have heated centrifuges, as do milk plants using official methods.^{1*} Higher results are obtained with heated centrifuges.

If both the milk plant and association testers worked under the same conditions and both used standard procedures, their results would probably agree closely.

What may cause a faulty Babcock test of milk?

1. Improper preparation and mixing of the sample
2. Inaccurate sampling
3. Charred fat columns
4. Curdy fat columns
5. Using water with a high mineral content
6. Not enough centrifuging
7. Extreme fluctuations in temperature of water bath
8. Inaccurate reading of the test

MODIFIED BABCOCK PROCEDURES FOR OTHER MILK PRODUCTS

The regular Babcock method gives reliable results with milk and cream but not with most other milk products. The sulfuric acid used in the test acts on the sugar in chocolate milk, sweetened condensed milk, and ice cream and causes the fat column to be so dark that it cannot be read accurately. We do not know why the regular Babcock method fails to give an accurate test with evaporated milk, homogenized milk, skimmilk, and buttermilk. The difficulty may be associated with the phospholipids and with the fat emulsion.

An accurate fat test is essential for homogenized milk, chocolate milk, evaporated milk, sweetened condensed milk, and ice cream, because these products must conform to specified fat standards. It is essential for skimmilk, buttermilk, and whey in order that the efficiency of skimming, of butter making, and of cheese making can be determined.

The modified Babcock methods are useful because they employ the Babcock test equipment and results can be obtained quickly.

Homogenized Milk

Several modifications of the Babcock method give satisfactory results with homogenized milk. The essential difference from the regular Babcock method is in the way the acid is added. In the modified methods the acid is added in three different portions and the bottles are agitated after each addition of acid. The method given here is that described by Lucas and Trout.^{21a*, 30*}

Preparing samples, pipetting. Proceed as for whole milk, being careful not to mix air into sample.^{6*}

Adding acid. Have acid and milk at 70°. Add a total of 17.5 ml. of sulfuric acid with a specific gravity of 1.82-1.83. Add this acid in three different portions: 8, 5, and 4.5 ml. respectively. Shake bottles with a rotary motion each time after adding the acid and for at least 15 seconds before adding the second and third portions of acid; then shake bottles for 2 to 3 minutes in a mechanical shaker.

Centrifuging. Whirl bottles for 5 minutes. Add water at 135°-140° to about $\frac{1}{4}$ inch below base of neck. Shake bottles 5 to 10 seconds. Then proceed as for regular Babcock method (see page 42).

There is another modification of the Babcock test which has given satisfactory results with homogenized milk. It differs from the modification described above only in the way the acid is added.

Adding acid. Have acid and milk at 70°. Add acid to test bottle in two different portions of 8.5 ml. each. As first portion of acid is added, place each bottle on the shaking device, which is in motion (Fig. 4). When first portion of acid has been added to the last bottle, add second portion of acid to the bottles in the same order as the first portion, with the shaking device in motion.

Chocolate Milk

The Pennsylvania method^{25*} is used for testing chocolate milk. The chemicals needed are ammonium hydroxide, containing 28-29 percent ammonia; normal butyl alcohol with a boiling point of 242.6 (117° C.); and sulfuric acid with a specific gravity of 1.72-1.74. To prepare the acid, add $3\frac{1}{2}$ parts (by volume) of commercial sulfuric acid (with a specific gravity of 1.82-1.83) to 1 part of water. (*Caution: Only a person who has had the required training in chemistry should make this mixture.*)

Preparing samples, pipetting. Proceed as for whole milk.

Adding reagents. Add 2 ml. of ammonium hydroxide from a buret and mix for about $\frac{1}{2}$ minute. Add 3 ml. of normal butyl alcohol from a buret and mix for 2 minutes. Add 17.5 ml. of sulfuric acid with specific gravity of 1.72-1.74 and shake until digestion is complete, as indicated by a dark color.

Centrifuging. Proceed as for whole milk.

Reading fat column. Put bottles in a water bath as for Babcock procedure. Immediately before reading each test, add 2 to 3 drops of meniscus remover (glymol), allowing it to flow down

inside the neck. With calipers, measure fat column from its lowest point to line between glymol and fat. Record results.

Evaporated Milk

The various tests for evaporated milk have been investigated by a committee of the American Dairy Science Association. The committee recommends both the Pennsylvania method^{25*} and the Minnesota method.^{27*} Both methods give results that agree closely with the Mojonnier method.

Pennsylvania method. *Preparing and sampling.* Heat sample to 95°-100° and mix by pouring it 6 times from one container to another. Weigh 6 grams into either an 8-percent milk test bottle or into a 20-percent ice-cream test bottle.

Procedure. Proceed as for chocolate milk (page 52). Multiply fat reading by 3 when using milk test bottle and by 1½ when using ice-cream test bottle.

Minnesota method.^{4*} *Preparing and sampling.* Heat sample to 95°-100° and mix by pouring it 6 times from one container to another. Weigh 9 grams of sample into a 20-percent ice-cream test bottle.

Adding reagent. An alkaline reagent, instead of sulfuric acid, is used to dissolve the solids-not-fat and release the fat. This reagent consists of sodium carbonate, sodium salicylate, sodium hydroxide, and alcohol. Add 15 ml. of reagent to each test bottle. Shake for ½ minute.

Heating. Unlike sulfuric acid, the alkaline reagent does not produce heat when added to milk products. The test bottles must therefore be placed in hot water to dissolve the solids-not-fat. Put bottles in a rack at least ½ inch above bottom of water bath at 212° and hold them at this temperature 12 to 15 minutes.

Shaking. Shake test bottles vigorously when at least half the contents have turned dark brown. This color will appear within 2½ minutes. After about 1 minute shake bottles again. Be sure to do this carefully, as otherwise the contents may boil thru neck of bottle and ruin test.

Centrifuging. Place bottles in centrifuge and whirl them $\frac{1}{2}$ minute. Add water at 135° - 140° to raise fat into graduated neck of bottle. Whirl bottles $\frac{1}{2}$ minute.

Reading fat column. Place bottles in a water bath at 135° - 140° for 5 minutes with water slightly above level of fat columns. Before removing each bottle, add to it 2 to 3 drops of meniscus remover (glymol) allowing it to flow down inside the neck. Measure fat column with calipers from its lowest point to fat-glymol line. Record results.

Sweetened Condensed Milk

A committee of the American Dairy Science Association^{4*} has recommended the Minnesota and Pennsylvania methods for testing this product. The results agree closely with those obtained by the Mojonnier method.

Preparing and sampling (both methods). The added sugar makes sweetened condensed milk very viscous. Water is added to the condensed milk so that it will be easier to mix and weigh it into the test bottles.

To add water, balance one beaker on each pan of a cream scale or on any other scale that is equally sensitive. Very carefully weigh about 100 grams of sweetened condensed milk into one beaker and enough distilled water into the other beaker to balance the scales. Mix contents of beakers well and place in a stoppered flask.

Procedure for Minnesota method. Weigh 9 grams of prepared sample into either a 20-percent ice-cream test bottle or an 8-percent milk test bottle. Proceed with test as outlined on page 53. Multiply fat reading by 2 when using ice-cream test bottle and by 4 when using milk test bottle.

Procedure for Pennsylvania method. Weigh 9 grams of prepared sample into either a 20-percent ice-cream bottle or 8-percent milk test bottle. Proceed with test as outlined on page 52. Multiply results with the ice-cream test bottle by 2 and with the milk test bottle by 4.

Skimmilk

A separator in good mechanical condition will produce skim-milk with a fat content of about .06-.08 percent as indicated by the Mojonnier method.^{15*} The plant operator should make fat determinations on skimmilk daily in order to check the skimming efficiency of the plant. Farmers will find it worth while to have their separators checked at regular intervals by dairy herd improvement association testers or creamery testers, or by any laboratory capable of rendering this service; otherwise they may lose a lot of fat over long periods without realizing it.

Sampling. To observe the efficiency of skimming over a period of time, take sample of skimmilk direct from the separator in the plant at regular intervals, at least every 30 minutes. Take not less than 75 ml. of skimmilk and place in a composite bottle that can be tightly stoppered. Hold sample at 35°-40° and test within 24 hours.

American Association test. This method is recommended for skimmilk by a committee of the American Dairy Science Association.^{12*} The chemicals used in this test are normal butyl alcohol with a boiling point of 242.6° (117° C.) and sulfuric acid with a specific gravity of 1.82-1.83. Following are the steps in making this test:

1. Add 2 ml. of normal butyl alcohol from a buret to each test bottle. Do not add alcohol with a pipet.
2. Heat sample to 95°-100° and mix by pouring from one container to another at least 4 times.
3. Add 9 grams of sample to test bottle and mix contents well.
4. Add 7 to 9 ml. of sulfuric acid and mix contents well.
5. Whirl 6 minutes with test bottles arranged in centrifuge with graduated necks toward outside, and add water at 135°-140° to base of bottle neck.
6. Whirl 2 minutes and add water at 135°-140° to bring fat into neck of bottle.
7. Whirl 2 minutes and place bottles in a water bath at 135°-140° for 5 minutes.
8. Multiply actual reading by 2 because only half the capacity of bottle was utilized.

Buttermilk

The fat content of buttermilk is affected by the season of the year, the fat content of the cream, temperature of pasteurization, time and temperature of cooling cream before churning, acidity, and churning temperature. A certain amount of fat is always found in buttermilk as a result of the churning process, and this cannot be avoided with even the most efficient methods, but buttermilk should not ordinarily have a fat content of more than .6 percent by the Mojonnier method. (See prominent textbooks^{17,28*} on the manufacture of butter.)

Sampling. Sample buttermilk after it starts to drain from the churn.^{17*} Do not include granules of butter in sample, as they should be retained by the screen at gate of churn. Take not less than 75 ml. of buttermilk and place in a composite bottle that can be tightly stoppered. Hold sample at 35°-45° and test within 24 hours.

American Association test. This method for testing buttermilk is recommended by a committee of the American Dairy Science Association.^{12*} See page 55 for directions.

Whey

Loss of fat in whey is affected by several conditions related to the making of cheese. These conditions are discussed in prominent books on the manufacture of cheese.^{31, 36*} The fat content of whey will always average about .3 percent, as no practical method has been found for preventing this amount of loss. Most of the loss can, however, be recovered by passing the whey thru a centrifugal separator.

Sampling. The sampling of whey for fat is more complicated than the sampling of other products because the samples must be taken at three different stages in the manufacturing process in order to obtain an accurate accounting. Following is a method suggested by one investigator.^{24*}

1. Take sample of whey from vat immediately after curd and whey have been stirred and just before whey is to be drained out.

2. After main portion of whey has drained out, there is a period when whey continues to drain slowly from curd. Save all this whey up to time curd is pressed. Mix well and obtain a sample.
3. When cheese is placed in press, some whey is removed. Save this portion also. Mix well and take a sample.

To calculate the amount of whey removed from the vat the first time, subtract from the weight of the milk in the vat the sum of the weights of curd removed from the press, the later drainage of whey from the curd in the vat, and the drainage of whey from the press.

A test of the whey in the vat (the first sample taken) may give enough information for routine operations. If so, estimate the amount of whey by subtracting the yield of cheese from the weight of milk.

American Association test. This method is recommended by a committee of the American Dairy Science Association^{12*} for whey from Cheddar, Blue, Edam, and Swiss cheese. For directions see page 55.

Pennsylvania test. This test is suggested for whey from Cheddar, Blue and Edam cheese. Proceed as for chocolate milk (page 52).

Ice Cream: Nebraska Modification

Reagents. The Nebraska test for ice cream employs two reagents, which are designated as A and B. *Reagent A* contains 90 ml. of normal butyl alcohol and 10 ml. of chemically pure ammonium hydroxide. This reagent must be kept in a tightly stoppered bottle.

FIVE MODIFICATIONS of the Babcock method for testing ice cream for fat have been developed and are described here. These are the Pennsylvania,^{25*} Minnesota,^{27*} Nebraska,^{5*} and Illinois^{23*} methods and one known as the glacial acetic-acid method. None of these methods have yet been approved by official organizations and should therefore be regarded as tentative.

Reagent B contains 100 ml. of sulfuric acid (with specific gravity of 1.82-1.83) and 100 ml. of ethyl alcohol (95-percent pure or specially denatured). To mix *Reagent B* pour the sulfuric acid slowly into the alcohol. As the sulfuric acid will generate considerable heat, be sure to use a pyrex glass container and to add the acid slowly by running it down the side of the container. Pure alcohol is best because with it the reagent remains stable longer than with denatured alcohol. When denatured alcohol is used a light-brown color develops in a few days at room temperature. For this reason only a small amount of the reagent should be prepared when denatured alcohol is used.

Preparing sample. Place not less than $\frac{1}{2}$ pint of ice cream or ice-cream mix in a bottle that can be tightly stoppered. Heat contents to 95°-100° in water bath maintained at 100°-105°. Keep water level in bath about $\frac{1}{2}$ inch above level of ice cream.

Balancing scales. Proceed as for cream.

Mixing and weighing samples. Pour sample from one container to another at least 6 times. Transfer 9 grams of the sample with a pipet into an ice-cream test bottle or into an 8-percent milk test bottle.

Adding reagents. Add 5 ml. of *Reagent A* and mix thoroly by shaking. Add 30 ml. of *Reagent B* or slightly less (28-29 ml.) if necessary to keep liquid below bottle neck. Shake contents of bottle until all curd is completely dissolved.

Heating. Heat bottles in water bath at 175°-180° for 15 minutes and shake contents at least 3 times during this time.

Centrifuging. Whirl bottles for 5 minutes at normal Babcock-test speed. Shake them thoroly. If fat column is below neck, add water at not less than 180° to raise level to $\frac{1}{4}$ inch below base of neck. Whirl for 3 minutes. Shake bottles again if any curd appears. Add water at 180° to raise top of fat column to about the 7-percent mark. Whirl bottles for 1 minute.

Reading fat column. Place bottles in a water bath for 5 minutes at 135°-140°. Add glymol in same manner as for cream test. When using a milk test bottle multiply reading by 2. Read ice-cream test bottle directly in percent.

Ice Cream: Minnesota Modification

Preparing sample. Follow Nebraska method (page 58).

Balancing scales. Proceed as for cream.

Mixing and weighing samples. Follow Nebraska method (page 58).

Making the test. See page 53.

Ice Cream: Pennsylvania Modification

This method can be used for ice cream of any flavor. Nuts and fruit in the sample should be finely divided by using a mixer of the malted-milk type. Sample should be well mixed while it is being weighed.

Preparing and weighing samples. Follow Nebraska method.

Procedure. Follow instructions for Pennsylvania method for chocolate milk on page 52. If milk test bottle is used, multiply fat ready by 2. If ice-cream test bottle is used, read fat test directly.

Ice Cream: Illinois Modification

Reagents. This method may be used with vanilla, fruit, and chocolate ice creams. In this method two alkaline reagents are employed:

Reagent A consists of —

75 ml. of C.P. ammonium hydroxide

35 ml. of N-butyl alcohol

15 ml. of 95-percent ethyl alcohol

Reagent B consists of —

200 grams of trisodium phosphate (commercial grade)

150 grams of sodium acetate (commercial grade)

1 liter of water

Reagent B will remain in solution at room temperature, but the salts will begin to crystallize out at 50°-60° and they must be dissolved by heating in a water bath before the reagent can be used.

Preparing and weighing samples. Follow Nebraska test (page 58).

Adding reagents. Add 2.5 ml. of Reagent A from a buret

or pipet. Mix thoroly. Add 9 to 10 ml. of Reagent B with a graduated cylinder and mix thoroly.

Heating. Place test bottles in a water bath so that water level extends slightly above their contents, and heat to boiling. Keep at boiling point 4 to 5 minutes. Shake bottles well 2 or 3 times while heating. Continue heating until fat separates on surface and forms a clear layer: this will take 15 to 20 minutes for plain ice cream and 30 to 45 minutes for chocolate ice cream.

Centrifuging. Whirl bottles for 5 minutes and add water at 135° - 140° to base of neck. Whirl for 2 minutes and add water to bring top of fat column to about 7-percent mark. Whirl for 1 minute.

Reading fat column. Put bottles in water bath at 135° - 140° for 5 minutes. Read fat from bottom of column to top of upper meniscus.

Ice Cream: Glacial Acetic and Sulfuric Acid Modification

Preparing sample. Follow Nebraska method (page 58).

Balancing scales. Proceed as for cream.

Mixing and weighing samples. Follow Nebraska method (page 58).

Adding reagents. Add 9 ml. of water. Then add 13 ml. of glacial acetic acid and mix well. Finally add 13 ml. of sulfuric acid (specific gravity of 1.82-1.83) in about three equal portions, shaking sample after each addition.

Centrifuging. Proceed as for milk.

Reading fat column. Proceed as for milk. If an 18-gram bottle is used, double the reading.

OTHER TESTS FOR MILK AND MILK PRODUCTS

Determining Total Solids

Accurate determination of the total solids in milk and milk products is frequently required. Such a determination, along with other tests, makes it possible to control the composition of these products and make sure that they comply with specified standards as they pass into the channels of trade.

Equipment needed. An inexpensive method for determining total solids in milk and milk products has been developed by Livak and Doan^{21*} and Doan and Livak.^{7*} An apparatus known as the Dietert moisture teller is used (Fig. 16).

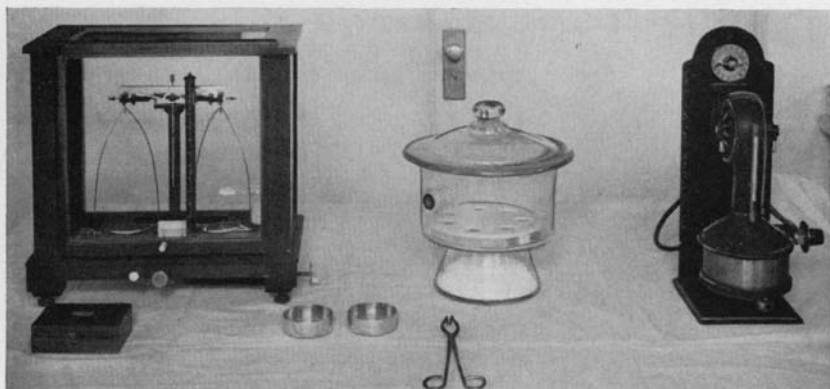
Caution: Only a technician who has been taught to do accurate work and who will observe all precautions in weighing, handling, and cooling dishes, samples, and residues, can use the Dietert apparatus for determining total solids.

Besides the Dietert moisture teller, the following equipment is needed: flat-bottom metal dishes 75 mm. in diameter (Mojonnier solids dishes with covers are satisfactory), a laboratory desiccator (dryer) of the proper size, an analytical balance, an accurate set of weights, and a pair of tongs made of metal that does not corrode.

Milk. Heat flat-bottom dish of 75-mm. diameter, with cover, in the Dietert apparatus for at least 10 minutes. During this time check and adjust the temperature of the apparatus. Cool the dish to room temperature and weigh it accurately on an analytical balance. Place about 2 grams of well-mixed milk in dish. Weigh quickly and accurately. Place dish in center of pan of Dietert apparatus. Spread milk evenly over bottom of dish. Check temperature again and dry for 20 minutes at 248° (120° C.).

When milk is dried, transfer dish immediately to desiccator and cool to room temperature. Weigh dish and dried residue accurately. The results are expressed as a percentage of total solids. For example, if the sample of milk weighs 2.1345 grams and the dried residue weighs .2647 gram, the total solids will be

$$12.4 \text{ percent: } \frac{.2647}{2.1345} \times 100 = 12.4.$$



Equipment required for making total-solids determinations. Left to right: balance and weights, solids dishes, tongs, desiccator, and Dietert moisture teller. The Dietert apparatus is tube-shaped, with a flared enlargement at the bottom. The upper part of the tube contains an electric blower and heating element. A removable dishholder with a fine brass screen is held by a spring clamp against the flared end of the tube. Above the dishholder is an air diffuser and a thermometer. An automatic timing dial and switch are located in the upright panel. The dial is graduated in half-minute intervals from 0 to 15. A thermo-regulator is attached to the right side of the flared base—temperature can be adjusted by turning a knob. Operation of this moisture teller takes skill and experience.

(Fig. 16)

Skimmilk. Proceed as for milk.

Cream. Proceed as for milk except dry the sample in the Dietert apparatus for 25 minutes at 248° (120° C.).

Condensed milk, condensed skimmilk, and evaporated milk. Proceed as for milk except weigh accurately 1 gram of the product, dilute with 1 ml. of distilled water, and dry for 25 minutes at 248° (120° C.).

Ice-cream mix. Proceed as for milk except weigh about 1 gram of the mix accurately into an aluminum dish and dilute with 1 ml. of warm water. Spread diluted mix evenly over dish. Heat sample for 30 minutes at 266° (130° C.).

Sweetened condensed milk. Proceed as for ice cream except add 2 ml. of water at about 130° and dry the sample for 20 minutes at 302° (150° C.).

Cool the empty dishes, samples, and residues to room temperature and weigh.

Determining Specific Gravity of Milk

The specific gravity of the mixed milk from a herd of cows is very constant, varying only slightly thruout the year. Knowledge of the specific gravity of milk is useful in identifying samples suspected of having been diluted with water or of having been partly skimmed. The method is not accurate enough to detect the amount of water added; but when used with the fat test, it enables an operator to find the suspected samples. If the weight, fat test, and specific gravity determinations of any patron's milk all vary greatly from day to day, the creamery operator has reason to suspect that the composition of the milk is being altered. A freezing-point determination made with the Hortvet cryoscope will show whether water has been added to the suspected samples.

Specific gravity determinations of milk can be made with a lactometer (Fig. 17). A lactometer operates on the same principle as the hydrometer used to determine the strength of the acid in an automobile battery. Three general types of lactometers are in use:

The Quevenne lactometer is the type most commonly used. It is calibrated in units from 15 to 40. These units correspond to specific gravities of 1.015 to 1.040. This lactometer has a thermometer with 2 degrees Fahrenheit for each division.

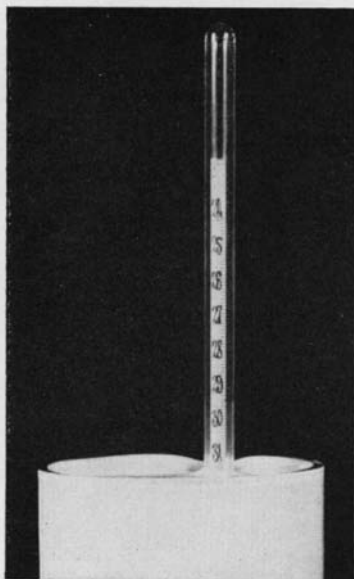
The U. S. Department of Agriculture lactometer is the most accurate. It is calibrated in units from 24 to 37 in divisions of $\frac{1}{10}$ degree. These divisions correspond to specific gravities of 1.024 to 1.037. This lactometer is recommended for routine dairy-plant use.

The New York Board of Health lactometer is calibrated in units of 0 to 120 degrees, zero corresponding to a specific gravity of 1.000, and 100 degrees corresponding to specific gravity of 1.029. This lactometer is not extensively used.

All lactometers should be tested for accuracy before they are used. An accurate thermometer graduated in intervals of $\frac{1}{10}$ degree Fahrenheit is needed in order to obtain the correct temperature for specific gravity determinations.

Taking sample. Observe same care as in obtaining a representative sample for Babcock test.

Preparing sample. The sample must consist of at least 1 pint of milk. Adjust temperature of sample to 113° and hold at this temperature for $\frac{1}{2}$ minute. Cool to slightly above 86° . Mix sample by pouring it 3 times from one container to another. Be careful to pour it against walls of container to keep from mixing air into it. Foam on surface of sample will interfere with lactometer reading.



Lactometer and cylinder for determining specific gravity of milk. This type of lactometer is recommended for routine dairy-plant analysis. It is calibrated in units from 24 to 37 in divisions of $\frac{1}{10}$ degree. Note position of lactometer in cylinder of milk. Unless the cylinder is full of milk, it is hard to obtain an accurate reading. (Fig. 17)

Making determination. Be certain that milk in sample is at 86° . Fill a tall cylinder (Fig. 17) by pouring milk carefully against walls. Place lactometer in milk but do not drop it in, as this will wet more of the graduated scale than is necessary and will interfere with final reading. For accurate reading, the cylinder must be full when the lactometer floats in the milk.

When lactometer reaches a stationary position (Fig. 17), take reading at top of meniscus on stem. Record reading.

Calculating results. The total solids can be calculated according to the method of Sharp and Hart^{24a*} with Herrington's^{15a*}

correction. The formula follows:

$$\text{Total solids} = 1.2537 F + \frac{268 (Q + 3)}{Q + 1000}$$

F = fat percentage in whole numbers

3; 268; 1000; and 1.2537 = constants obtained by comparison with gravimetric methods of making total solids determination

Q = Quevenne or U.S.D.A. lactometer reading in degrees.

To apply this formula, assume, for example, that a sample of milk has 3.7 percent fat and a lactometer reading of 27.1. Using these values in the formula, we have:

$$\begin{aligned} \text{Total solids} &= 1.2537 \times 3.7 + \frac{268 (27.1 + 3)}{27.1 + 1000} \\ &= 4.63869 + \frac{268 \times 30.1}{27.1 + 1000} \\ &= 4.63869 + \frac{8066.8}{1027.1} \\ &= 4.63869 + 7.853957 \\ &= 12.49 \text{ percent.} \end{aligned}$$

To obtain the specific gravity of a sample of milk from a New York Board of Health reading, it is necessary to change the reading to that of the Quevenne lactometer. Multiply the reading by .29 and prefix the figure 1.0. To change the Quevenne reading to the Board of Health reading, divide the Quevenne reading by .29. Both the Quevenne and the U. S. Department of Agriculture lactometers are read in the same manner, but the latter can be read more accurately.

An apparatus known as the Westphal balance gives a more accurate determination of specific gravity than does a lactometer, but a reliable lactometer is accurate enough for most routine plant work.

Analyzing Hard Cheese

A method for analyzing cheese of the hard types for fat, moisture, and salt has been approved by a committee of the American Dairy Science Association.^{33*} This method, with improvements by Wilster,^{36*} follows:

Taking sample. Cut a narrow, wedge-shaped piece that reaches from outer edge to center of cheese. Remove rind. Divide piece into strips and pass it thru a sausage grinder three times. If cheese cannot be cut, take a sample with a cheese trier. If only one plug of the cheese can be taken, cut it perpendicular to surface of cheese at a point $\frac{1}{3}$ the distance from the edge to the center, extending either entirely or half-way thru it. If possible, draw three plugs perpendicularly, one from center, one an inch from outer edge and halfway between the other two. Include rind with sample. Grind plugs with a sausage machine or, preferably, cut them fine with a vegetable ricer and mix thoroly. Put samples in small, stoppered, airtight containers immediately and analyze as soon as possible. Samples not analyzed at once should be kept in a refrigerator.

Determining fat. The method described by the Association of Official Agricultural Chemists* is recommended for regulatory laboratories, where a high degree of accuracy is required to determine compliance with state and federal standards.

The Babcock method^{33*} can be used for routine analysis in the cheese factory. Proceed as follows:

1. Weigh 9 grams of prepared sample into a dry, tared, 9-gram, 50-percent, large-bodied Babcock test bottle. Do this quickly to keep moisture from evaporating from sample.
2. Add about 12 ml. of water and mix well with cheese. Place bottle in a boiling-water bath for 5 to 10 minutes and mix occasionally. Cool contents to about 100°. If testing a number of samples, place bottles in a water bath and heat to 170° for about 15 minutes and cool bath to 150°.
3. Add carefully, in about three equal portions, 15 to 17 ml. of sulfuric acid of specific gravity 1.82-1.83, and shake after each addition of acid. When all particles have dissolved, add water at 160°-180° to bottom of neck.
4. Whirl at proper speed in a heated centrifuge for 5 minutes. Add water at 140° to raise fat into neck of bottle. Whirl for 3 minutes.
5. Place bottles in a water bath at 135°-140° for 5 minutes with water level above level of fat. Add glymol (meniscus remover) to each bottle as you read it. Duplicates should check within .5 percent.

Determining moisture

1. Dry moisture dishes and their covers for 1 hour at 212° (100° C.). Cool for $\frac{1}{2}$ hour in a desiccator (dryer) that contains calcium chloride or some other suitable nonliquid drying agent.
2. Weigh each dish and its cover on an analytical balance, preferably the chainomatic type. Record weight.
3. Put about 2 grams of cheese into dish. Replace cover immediately. Weigh accurately and record weight. Make analysis in duplicate or triplicate.
4. Dry samples with their covers off, in an oven at 212° (100° C.). Drying in a vacuum oven at 23 to 26 inches, for 4 to 6 hours, will bring the cheese to constant weight. If a vacuum oven is not used, dry for 24 hours. To reduce spattering, place samples in oven when temperature is below 122° (50° C.) so that they will heat slowly. Heat samples at 212° for $\frac{1}{2}$ hour before applying vacuum. Apply vacuum slowly and release slowly.
5. Put dishes in a desiccator for $\frac{1}{2}$ to 1 hour, or until they reach room temperature. Weigh each dish as quickly as possible and record weight.
6. Divide loss of weight by weight of sample and multiply by 100 to get percentage of moisture in cheese.

For routine laboratory analysis, a balance with a tare beam and with beams for direct reading should be used. The balance should have a sensitivity reciprocal of 15 milligrams (meaning that the pointer will move at least one division on the graduated scale when a 15-milligram weight is added to either pan while the scale has a full load and is balanced). A light-weight aluminum dish 50 mm. in diameter and 20 mm. deep should be used. It should have a loosely fitting inverted lid. Both dish and lid should be plainly marked. The procedure is as follows:

1. Weigh exactly 5 grams of freshly prepared sample into dish.
2. Place cover loosely on dish. Dry in an oven as mentioned above. Cool dishes with covers in desiccator and weigh. Read to nearest .1 percent on beam and multiply by 2. On duplicate samples moisture should check within .2 percent.

Determining salt

1. Weigh accurately 3 grams of cheese into a 300-ml. Erlenmeyer flask.
2. Add 10 ml. silver nitrate solution (29.06 grams Ag NO_3 per liter) or more than enough to combine with all the chlorine.

3. Add 15 ml. of halogen-free nitric acid (C.P.).
4. Add 50 ml. of distilled water.
5. Repeat Steps 1, 2, 3, and 4, but use sucrose instead of cheese.
6. Place flasks on a hot plate. As mixture boils, add 15 ml. of potassium permanganate solution (saturated) to each flask in approximately three equal portions.
7. After digestion, dilute to 100 ml.
8. Decant off clear liquid.
9. Wash precipitate with another 100 ml. of water and again decant.
10. Add 3 ml. indicator (saturated ferric ammonium sulfate).
11. Titrate against standard potassium thiocyanate solution (16.63 grams per liter) to first lasting change in color. Color should last about 15 seconds.

Example of calculation for determining salt

5.3 ml. = amount of potassium thiocyanate used for cheese

9.8 ml. = amount of potassium thiocyanate used for sugar

Since this is a back titration, subtract above amounts from 10 ml. to obtain amount of silver nitrate used:

Cheese sample: $10 - 5.3 = 4.7$ ml. silver nitrate

Sugar blank: $10 - 9.8 = .2$ ml. silver nitrate

By subtracting .2 ml. from 4.7 ml. we get 4.5 ml. as the corrected amount of silver nitrate used. (The .2 ml. disappears in the blank determination and cannot be accounted for.) Each ml. of silver nitrate is equal to .01 gram of salt. The percent of salt can therefore be calculated as follows:

4.5 ml. silver nitrate $\times .01 = .045$ gram salt

$\frac{.045}{3} \times 100 = 1.5$ percent, the amount of salt in sample.

Analyzing Butter

Butter can be analyzed for fat, moisture, salt, and curd by the Kohman method.^{20*} This method is practical for factory use and the results agree closely with the official method.^{1*} Anyone setting up a laboratory for butter analyses should read Wilster's^{34*} bulletin for a description of equipment which aids greatly in analyzing butter. The Kohman method, with refinements in equipment and technic as indicated by Wilster,^{34*} is described here.

Equipment and chemicals needed. Following is a list of the equipment and chemicals for making an analysis of butter.

Water bath. A water bath is useful for warming butter samples before they are stirred. The most efficient kind of water bath is heated by electricity and controlled by a thermostat.

Stirring device. A stirrer is useful when a large number of samples are analyzed. Each sample can then be thoroly mixed in a few seconds.

Weighing balance. Balance should have a sensitivity reciprocal of 15 milligrams. Altho the balance may be of either the beam or torsion type, it should preferably have 4 beams^{34*} so that the percentages of moisture and fat can be read independently of each other. The graduated beams should be kept clear and legible. If beams become dull, scrape away old paint, clean with petroleum ether, and apply white paint. Rub off wet paint with a cloth. Vertical markings on the beams will remain white. Set balance level on a firm support and protect it from drafts (Fig. 8). A plastic cover to protect balance against insects is recommended.

Cup for test. There are a number of aluminum cups that may be used. A satisfactory cup is $2\frac{7}{8}$ inches high, 2 inches in diameter at bottom, and $2\frac{3}{8}$ inches in diameter at top. It weighs about 21 grams and has 150 ml. capacity to overflowing. Top is flared and has a prominent pouring lip.

Heating device. An electric hot plate with a heating unit that will attain a temperature of 680° - 700° at the surface is satisfactory. It should be equipped with a three-heat switch. *An open flame is not safe.*

Cooling surface. The warm cups may be: (1) cooled to room temperature on a metal surface, (2) cooled in a desiccator, or (3) cooled in dry compartments surrounded by cold water. Method 3 takes the least time.

Tongs. Several pairs of crucible tongs made of noncorrosive metal are needed to handle aluminum beakers.

Weights. An accurate 10-gram weight must be used. This weight should be of same construction as those prescribed for the Babcock test for cream and should have same relative tolerances of accuracy. An extra weight that is known to be accurate should be kept on hand.

Sampling devices. Several metal spatulas and triers are satisfactory for sampling butter. Spatulas should have a 4-inch blade of stainless steel. A straight-sided $\frac{1}{2}$ -pint glass container tightly fitted with a screw cap and lined with paraffined cardboard is recommended when samples are kept for a short time.

Silver nitrate solution. The most convenient silver nitrate solution to use for the salt test contains 29.06 grams of silver nitrate per 1000

ml. This solution can be made up and checked for strength by a person who has had the necessary training in chemistry, or it can be bought from dairy supply firms.

Petroleum ether. This fat solvent should have a specific gravity of .634 to .660 at 77° and an initial boiling point of 120° to 140°.

Potassium or sodium-chromate indicator. A 10-percent solution is required for the salt test. It can be made by dissolving 10 grams of potassium dichromate in 90 grams of water, or it can be bought from dairy supply firms.

Glassware. A 50-ml. buret with supporting stand, or a buret attached to a flask, is needed for the salt test. Also needed are: four 250-ml. volumetric flasks; six 25-ml. pipets; three white cups; six plain glass stirring rods about 6 inches long with rubber tips. Rubber tips should be replaced when they show signs of wear.

Distilled water. As distilled water should be used whenever water is needed for the analysis of butter, a liberal supply should be available at all times. A satisfactory apparatus for distilling water can be bought from several chemical supply companies.

Sampling from churn. A more representative sample can be obtained by taking samples with a trier from several places in the churn.^{26*} Be sure to take sample where there is no free moisture. Scrape butter off trier with spatula and put it in sample jar. Close jar tight. Do not include butter that is on back of trier.

Sampling from tub or box. When taking a sample from a tub, insert trier practically its full length from top edge of tub. When sampling a box, insert trier at corner of the box and run it diagonally across and down to the corner opposite the point of entry. Give trier one complete turn and withdraw a full core. Place point of trier over mouth of sample jar and, with spatula, immediately scrape off a plug of butter in about 3-inch portions. Leave a portion about 1 inch long to fill the hole from which the plug was removed. Take two other trierfuls at points equally distant from the first and add to the sample jar in the same manner. Do not include moisture which adheres to outside of trier.

Clean trier and dry it with absorbent paper each time before drawing the next plug. Have trier at room temperature when sampling butter above 32°. When sampling butter stored below

32°, use a trier heated to 125°. If butter is frozen so hard that it cannot be sampled, temper it in a room at 35°-40° for 24 hours.

When tubs or boxes are marked with churn numbers, take samples as follows: sample one tub or box from each churning of 1 to 9 tubs or boxes; sample two tubs or boxes from each churning of 10 to 14 tubs or boxes. The samples from each tub or box from the same churning must be put together to make up a composite for that churning.^{1*}

If tubs or boxes are not marked with churn numbers, find the square root of the number in the lot and sample that number of tubs or boxes, but limit the number to a minimum of 3 and a maximum of 25.^{1*}

Sampling from prints.^{1*} Find the square root of the number of cases in the lot of butter to be sampled. Take one print each from that number of cases, but limit the number to a minimum of 5 cases or a maximum of 25. If the square root includes a fraction, disregard the fraction and sample an extra case.

When churnings can be identified, select cases to include each churning. If there are less than 5 cases in the lot, take a sample from each. Remove wrappers and put prints in separate containers. If prints weigh a pound or more, you may quarter each print and take 2 alternate quarters as the sample. With 4- and 8-ounce prints, take whole print as the sample.

Preparing samples for analysis. 1. *By shaking.*^{1*} Soften entire sample to 95° F. in a water bath at 102.2°, or heat to 95° in a constant-temperature oven held at 95°. Shake sample vigorously until it is a homogeneous semisolid mass. Weigh sample for analysis at once, using a spatula for stirring sample and weighing it into cup. If sample is kept for any length of time, soften it and shake it again before taking portion for analysis.

2. *With mechanical stirrer.*^{1*} Soften entire sample to 77°-86°. Stir with electric food mixer of double-beater type with variable control to give a maximum speed of 1000 r.p.m. A malted-milk stirrer is also satisfactory. Agitate each sample for 2 to 3 minutes, using an up-and-down movement and at the same time move container to improve thoroughness of mixing. Remix if weighing out sample for analysis is delayed over 3 hours or if temperature goes above 86° or below 73°.

Preparing balance for weighing. The balance may be adjusted properly while the samples are being brought to the proper temperature. Place the balance permanently on a firm and level table. Protect it from drafts (Fig. 8). Put the riders of beams of torsion scales at zero mark. Move large rider on tare beam to right as far as possible. Put small rider on tare beam on zero mark. Next, balance scales by adjusting leveling screws. Leave balance in same position until analysis has been completed. The modified torsion balance developed by Wilster^{34*} is convenient to use. It has 4 beams, 2 for fat and 2 for moisture. The 10-gram weight is not moved during the analysis and is the only loose weight used.

Determining moisture. Use a clean, dry weighing cup. Cup can be dried by heating it over a flame or on an electric heater and cooling to room temperature. Put cup on right-hand pan of scales and balance by first adjusting large tare rider and next the small rider. When cup is balanced, place weight on left pan and quickly weigh 10 grams of butter. Some moisture will be lost if weighing is not done rapidly. Air in the room should be reasonably dry.

Moisture is removed by heating cup and contents to about 300°. Heat by placing cup on an electric plate. Rotate cup during heating to hasten removal of moisture, reduce foaming, and prevent sputtering. Continue heating until butter ceases to foam and is medium-brown. This requires about 6 to 7 minutes.

Cool cup and moisture-free butter to temperature of the room. Cooling can be done most efficiently by placing cup in a metal compartment surrounded by a jacket of water. Do not cool in an open pan of water because water might splash into it. Move riders on two top beams until both sides balance. Read percentage of moisture to nearest $\frac{1}{10}$ of 1 percent.

Determining fat. Dissolve fat with petroleum ether. Add about 100 ml. of this solvent to the cup and contents. Stir with rubber-tipped glass rod. As salt and curd settle to bottom, scrape them to a point directly below the pouring lip so that you can pour off the ether without seriously disturbing the salt-and-curd mass. Let cup and contents stand for 3 to 4 minutes and then

pour off ether, being careful not to pour off any curd and salt. Again add about 100 ml. of solvent to remove last traces of fat. Let this stand 3 minutes. Then pour off ether.

Take care to retain materials in bottom of cup. Tap cup gently on table to distribute salt and curd over bottom. Remove ether that remains by placing cup on *electric hot plate*, never on an open flame unless it is covered with an asbestos screen. Heat cup and contents slowly at first; otherwise ether will evaporate so fast that small explosions will occur, and some curd and salt will be blown out of cup. At this point the residue of salt and curd should be powder-like and have a light-tan color. (If this residue sticks to bottom of cup, it indicates that some fat remains and a third extraction with ether is needed.) Cool cup to room temperature. This can be done quickly by putting cup in a water-cooled metal container as indicated on page 69.

Leave riders on two upper beams where they were when the moisture readings were made. Read percent of fat in butter directly by moving two lower beams until scale is balanced. The lower beam has 10-percent graduations; the next above has 1-percent graduations which are calibrated into smaller divisions, each representing $\frac{1}{10}$ of 1 percent.

(The ether can be salvaged from both extractions and used again. To save ether, use a distilling apparatus such as that described by Wilster.^{34*} When large numbers of analyses are being made, this is very economical. For disposal of fat-ether solutions, see page 77.)

Determining salt. To the residue in the aluminum cup add about 50 ml. of distilled water heated to 120°-130°. Rotate cup to release the particles of curd and salt. Pour into a 250 ml.-volumetric flask. Add another 50 ml. of warm water to cup to rinse out last remaining curd and salt, and add this to the flask. Pour enough distilled water into flask to bring liquid up to the mark. Place glass stopper in flask and mix well. Put 25 ml. of this solution in a white cup and add 2 to 3 drops of the sodium or potassium chromate indicator. Titrate slowly with silver nitrate, stirring constantly until a faint orange-red color is obtained. If silver nitrate solution is of such strength that 1 ml. is equal to 1 percent of salt, then salt percentage can be read direct from buret. The 25 ml.-portion titrated represents the salt in 1 gram of butter.

The salt test is based on the chemical reaction between silver nitrate and sodium chloride (salt). The silver nitrate reacts with the sodium chloride and forms a white precipitate of silver chloride. A solution of potassium chromate is used as an indicator. As soon as all the salt has combined with the silver nitrate, the potassium chromate combines with the excess silver nitrate to form silver chromate, which is orange-red and easily recognized.

Determining curd. Add together the percentages of fat, moisture, and salt, and subtract total from 100. The difference is the percentage of curd in the butter.

Common sources of error in analyzing butter^{34*}

1. **Inaccurate sampling.** Sample was not representative of churning; loose moisture was present; or sample was taken from churn before butter was completely worked.
2. **Moisture evaporated from sample before sample was analyzed.** Container must be air-tight.
3. **Improper mixing of sample.** A thoro emulsion of fat and moisture cannot be made if butter is hard and leaky. Sample should be mixed at 88°-93°.
4. **Inaccurate scales.** An accurate analysis cannot be made if beams stick or steel bands in scales become rusty. Scales should be kept in a dry, clean place.
5. **Inaccurate weights.** Weights should be checked every month against accurate weights kept for this purpose.
6. **Scales not set properly.** Scales should be level on a table that is free from vibrations.
7. **Cup wet or too warm.** Cup should be dry and cooled to room temperature before butter is weighed into it.
8. **Cup and moisture-free sample were too warm.** Cool them to room temperature.
9. **Scales standing in a draft.** A draft will affect the reading on these sensitive scales.
10. **Air too moist.** An accurate moisture determination cannot be made in a room where there is steam. Weighings should always be done in a dry room.
11. **Moisture not expelled.** Heat sample until fat is golden brown. If sample is burnt, another analysis should be made.
12. **Sample is heated too rapidly.** Excessively high moisture readings will occur if sample is heated too rapidly without agitation, because some fat will sputter out.

TESTING LABORATORY AND CARE OF EQUIPMENT

The laboratory for testing dairy products should be a separate room located near the main office of the plant. It should be easily reached from the processing and manufacturing rooms. It should be dry and should have its own ventilating system, since too much moisture damages many types of sensitive equipment. The temperature of the laboratory should be as constant as possible and near 70°.

The laboratory should be orderly and well kept. Careful work is not likely to be done in one that is dirty and disorderly.

Tables in testing laboratories should be durable. They should be covered with material that is resistant to moisture, acids, alkalies, and salts. Both lead and glass resist moisture and chemicals. Lead makes a better covering than glass because it has a softer surface. There will be less breakage of glassware on a lead-covered table. Another satisfactory cover is an asbestos-like board. It is durable, inexpensive, and quite resistant to sulfuric acid and other chemicals.

Washing basins of the three-compartment type are best, as equipment can be washed in one compartment and rinsed in the other two. Basins should be supplied with hot and cold water.

Walls in the laboratory should be painted a light color. Although natural lighting should be used to the fullest extent, good artificial light should be provided.

Scales, weights, microscopes, and similar metal equipment should not be stored in the same compartment with acids such as nitric or hydrochloric or with alkalies such as ammonium hydroxide. The fumes from these chemicals will corrode the metal. They may even ruin expensive laboratory apparatus. One separate storage compartment should be provided for acids, one for other dry chemicals, and one for metal apparatus of the various types. Such apparatus should be stored when not in use.

All glassware and other equipment should be washed as soon as the work is finished. Pipets and test bottles are hard to

clean if not washed immediately after use. A rack is useful for draining, washing, and rinsing test bottles.

There are several ways to wash Babcock test bottles. The following procedures will give good results:

1. Shake the rack when the bottles are emptied. This will help to loosen the material that is stuck to the inner surfaces of the bottles.
2. Fill bottles with alkaline solution at 115° - 120° . While constantly shaking the bottles, empty the contents into the wash basin. Repeat. If bottles are not clean, repeat again. (*Do not leave bottles in washing solutions for long periods as the alkali may cause the glass to become opaque.*)
3. Rinse twice with hot water at 115° - 120° . At this stage the bottles will usually be clean, with bright surfaces that drain quickly. If a film persists after the first rinsing with hot water, rinse again with cold water.
4. Leave the bottles upside down to dry. A dry bottle should be clean and bright. (A piece of glassware is clean when it drains without drops of water clinging to the inner and outer surfaces.) If a small amount of wetting agent was used in the rinse solution, the bottles will drain faster.

Brushing the inside of test bottles is seldom needed if the bottles are immediately drained, washed, and rinsed correctly.

Washing compounds that contain large amounts of wetting agents are not satisfactory for cleaning test bottles because they leave a foam which is hard to remove. A 2-percent solution of trisodium phosphate is an excellent cleaning agent for Babcock glassware. There are other satisfactory cleaning compounds also.

Pipets should be cleaned by the same general procedure as test bottles, and placed in a pipet holder to drain.

The centrifuge should be kept in good mechanical condition, clean, and properly oiled. It should be checked frequently for correct speed. Centrifuges tend to operate at lower speeds as they are continued in service. When this stage is reached, a competent mechanic should repair the machine.

When test bottles break in centrifuge, as they sometimes do during the whirling process, the machine will usually start vibrating. Stop it at once. *Do not open the cover until the wheel has stopped*, as the glass and acid that has been released will be

thrown out and may injure you. Balance the wheel with a test bottle containing water, and complete the tests.

As soon as the tests are read, remove the cups, wash thoroly in a suitable alkaline solution, and rinse. (If acid spilled in the centrifuge from broken bottles is not promptly removed, vital parts may be damaged and the life of the machine may be shortened.) Remove broken glass from centrifuge, wash inner surface of the machine with same kind of alkaline solution as used for cups. Remove traces of the washing solution with a cloth or a sponge moistened with clean water.

When centrifuge is thoroly dry, wipe inner surface, including cups, with a cloth containing light oil. This will retard corrosion and make the machine last longer.

Centrifuges and other equipment made entirely or partly of aluminum require special care, as this metal is readily decomposed by strong alkalis. For this reason, do not use strong alkaline washing compounds to clean these machines or any other equipment made of aluminum. In fact, strong alkalis cause more or less corrosion on all plated metal surfaces.

Aluminum dishes used in total-solids determinations should be washed only with solutions recommended for this purpose.

How to dispose of fat-ether solutions is always a problem. Most city ordinances prohibit putting such inflammable materials into the sewer system. The safest way is to pour such a mixture into a separate container and empty it as soon as analyses are completed. The solution should always be dumped where it will not harm vegetation.

Disposal of acid from Babcock test bottles is also a problem. In some plants the acid is drained into the sewer system and thus diluted with large quantities of cold water. This method (according to the writer's observation) has been satisfactory in some plants over a period of years. But in one plant the acid decomposed the drain pipes and caused some damage to walls and furniture. Whether acid will harm the pipes probably depends on the construction, length, and slope of the pipes, the amount of water in the laboratory with which to dilute the acid, and the distance of the pipes from the main sewer line. The drainage

basin in the laboratory and the pipe leading from it to the sewer should be made of lead.

(It is best not to drain acid into the sewer without approval of an engineer and a regulatory official. Safest way is to drain bottles into a receptacle of glass or glazed crockery with a durable handle. Acid can then be carried out of plant and poured where it will do no damage. Do not pour where there is slightest chance that it will destroy plants or animals or damage property.)

TABLE OF EQUIVALENTS

Fahrenheit and Centigrade

Freezing point of water = 0° C. or 32° F.

Boiling point of water = 100° C. or 212° F.

An interval of 9° F. = an interval of 5° C.

To convert °C. to °F. multiply °C. by 9/5 and add 32.

To convert °F. to °C. subtract 32 from °F. and multiply by 5/9.

Weights and Measures

62.37 lb. = weight of 1 cubic foot of water at 60° F.

62.425 lb. = weight of 1 cubic foot of water at 39.2° F.

1.2 U.S. gallons = 1 imperial gallon

8.337 lb. = weight of 1 U.S. gallon of water at 60° F.

1.032 gm.^a = weight of 1 ml. of average milk at 60° F.

8.60 lb. = weight of 1 gallon of average milk at 60° F.

($8.337 \times 1.032 = 8.6037$ lb.)

1.036 gm. = weight of 1 ml. of skimmilk at 60° F.

8.64 lb. = weight of 1 gallon of skimmilk at 60° F.

($8.337 \times 1.036 = 8.6371$ lb.)

1.011 gm. = weight of 1 ml. of 20 percent cream at 60° F.

8.43 lb. = weight of 1 gallon of 20 percent cream at 60° F.

($8.337 \times 1.011 = 8.4287$ lb.)

.993 gm. = weight of 1 ml. of 40 percent cream at 60° F.

8.28 lb. = weight of 1 gallon of 40 percent cream at 60° F.

($8.337 \times .993 = 8.2786$ lb.)

1000 milliliters (ml.) = 1 liter

1.0000027 cubic centimeters (cc.)^b = 1 ml.

1 U.S. gallon = 3,785.332 ml. or 3.785332 liters

1 ounce (avoir.) = 28.3495 grams (gm.)

1 pound (avoir.) = 453.5929 gm. (453.6)

1 kilogram = 1000 gm. = 2.2046 lb.

1000 ml. of pure water at 39.2° F. (4° C.) = 1000 gm.

1 gram = 1000 milligrams (mg.)

^a 1.032 is thus the specific gravity of average milk at 60° F.

^b For practical purposes the units ml. and cc. may be considered equal.

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